MERCURY Acceptance Test Procedures and Specifications

MERCURY NMR Spectrometer Systems

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VARIAN

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for ***MERCURY*** NMR Spectrometer Systems

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SAFETY PRECAUTIONS

Observe the following safety precautions during installation, operation, maintenance, and repair of this instrument. Failure to comply with these precautions, or with specific warnings and cautions elsewhere, violates safety standards of design, manufacture, and intended use of the instrument. Varian assumes no liability for customer failure to comply with these precautions.

The following warning and caution illustrate the style used in Varian manuals for safety precaution notices and explain when each type is used:

WARNING: ***Warnings*** **are used when failure to observe instructions or precautions could result in injury or death to humans or animals, or significant property damage.**

CAUTION: ***Cautions*** **are used when failure to observe instructions could result in permanent damage to equipment or data.**

WARNINGS

Cardiac pacemaker and metal prosthetics wearers must remain more than 2.8 meters (9 feet) from the magnet system until safety is clearly established.

The ***MERCURY*** magnet system generates strong magnetic and electromagnetic fields that can affect operation of some cardiac pacemakers or harm a metal prosthesis. Pacemaker wearers should consult the user manual provided by the pacemaker manufacturer or contact the pacemaker manufacturer to determine the effect on a specific pacemaker. Pacemaker wearers should always notify their physician and discuss the health risks of being in proximity to magnetic fields. Wearers of metal prosthetics should contact their physician to determine if a danger exists. The following table may help determine the effect of a system on pacemakers or a metal prosthesis. The table shows the radial (i.e., horizontal) and axial (vertical) extent of the 5-gauss-level stray magnetic field as measured from the magnet center:

Proton Frequency (MHz)	*Bore (mm)*	*Radial Extent (m)*	*Axial Extent (m)*
200	54	1.50	1.50
200 long-hold	54	2.20	2.80
300	54	1.64	2.05
300 long-hold	54	2.20	2.80
400	54	2.20	2.80
400 long-hold	54	2.20	2.80

Refer to the ***MERCURY*** *Installation Planning Guide* for additional stray magnetic field plots and the effect of the stray field on electronic equipment. Varian provides signs containing this warning with each system. Post the signs according to the directions on the sign. Additional signs are available by request.

WARNINGS *(continued)*

Keep metal objects at least 2.8 meters (9 feet) away from the magnet.

The strong magnetic field of the dewar attracts objects containing steel, iron, or other "magnetic" materials, such as tools, electronic equipment, compressed gas cylinders, steel chairs, and steel carts. Unless restrained, such objects can suddenly fly towards the magnet, causing personal injury and extensive damage to the probe, the dewar, and the superconducting solenoid. Only nonferromagnetic materials (such as plastics, aluminum, wood, and stainless steel) should be used in the area around the magnet dewar.

Only qualified maintenance personnel shall remove equipment covers or make internal adjustments.

Dangerous high voltages exist inside the equipment that can kill or injure.

Do not substitute parts or modify the instrument.

Any unauthorized modification could injure personnel or damage equipment and potentially terminate the warrantee agreements and/or service contract. Written authorization approved by the ***MERCURY*** product manger of Varian, Inc. is required to implement any changes to the hardware of the spectrometer. Maintain safety features by referring service to a Varian service office.

Do not operate in the presence of flammable gases or fumes.

Operation with flammable gases or fumes present creates the risk of injury or death from toxic fumes, explosion, or fire.

Leave area immediately in the event of a magnet quench.

If the magnet dewar should quench (sudden appearance of gasses from the top of the dewar), leave the area immediately. Sudden release of helium or nitrogen gases can rapidly displace oxygen in an enclosed space creating a possibility of asphyxiation. Do not return until the oxygen level returns to normal.

Avoid helium or nitrogen contact with any part of the body.

In contact with the body, helium and nitrogen can cause an injury similar to a burn. Never place your head over the helium and nitrogen exit tubes on top of the magnet. If helium or nitrogen contacts the body, seek immediate medical attention, especially if the skin is blistered or the eyes are affected.

WARNINGS *(continued)*

On magnets with removable quench tubes, keep the tubes in place except during helium servicing.

Oxford 80-day hold 200/54 and 300/54 superconducting magnets (ALOX) include removable helium vent tubes. If the magnet dewar should quench (sudden appearance of gasses from the top of the dewar) and the vent tubes are not in place, the helium gas would be partially vented sideways, possibly injuring the skin and eyes of personnel beside the magnet. During helium servicing, when the tubes must be removed, follow carefully the instructions and safety precautions given in the Oxford documentation.

Do not look down the upper barrel.

Unless the probe is removed from the magnet, never look down the upper barrel. You could be injured by the sample tube as it ejects pneumatically from the probe.

Do not exceed the boiling or freezing point of a sample during variable temperature experiments.

A sample tube subjected to a change in temperature can build up excessive pressure, which can break the sample tube glass and cause injury by flying glass and toxic materials. To avoid this hazard, establish the freezing and boiling point of a sample before doing a variable temperature experiment.

Support the magnet and prevent it from tipping over.

The magnet dewar has a high center of gravity and could tip over in an earthquake or after being struck by a large object, injuring personnel and causing sudden, dangerous release of nitrogen and helium gases from the dewar. To prevent tip-over, at least two ropes (each rated at least twice the weight of a full magnet) should be suspended from the ceiling on either side of the magnet and firmly attached to the upper part of the magnet. The ropes must not be under tension, since this could transfer building vibrations to the magnet and affect NMR spectra. To calculate rope size and ceiling attachment points, refer to the site installation plan for the weight and height of the magnet. On 80-day hold 200/54 and 300/54 magnets (ALOX) only, an alternative is to bolt the magnet to the floor; however, floor mounting can be used only if it is first confirmed that floor vibration will not interfere with the operation of the spectrometer.

CAUTIONS

Keep magnetic tapes, credit cards, and watches away from the magnet dewar.

Most personal plastic cards, such as automated teller (ATM) and credit cards, contain a strip of magnetic media that can damaged by a strong magnetic field. Many wrist and pocket watches are also susceptible to damage from intense magnetism.

Check helium and nitrogen gas flowmeters daily.

Record the readings to establish the operating level. The readings will vary somewhat because of changes in barometric pressure from weather fronts. If the readings for either gas should change abruptly, contact qualified maintenance personnel. Failure to correct the cause of abnormal readings could result in extensive equipment damage.

Do not remove the relief valves on the vent tubes.

The relief valves prevent air from entering the nitrogen and helium vent tubes. Air that enters the magnet will contain moisture that can freeze, causing blockage of the vent tubes and possibly extensive damage to the magnet. Except when transferring nitrogen or helium, be certain that the relief valves are secured on the vent tubes.

Radio-Frequency Emission Regulations

The covers on the instrument form a barrier to radio-frequency (rf) energy. Removing any of the covers or modifying the instrument may lead to increased susceptibility to rf interference within the instrument and may increase the rf energy transmitted by the instrument in violation of regulations covering rf emissions. It is the operator's responsibility to maintain the instrument in a condition that does not violate rf emission requirements.

Chapter 1. Introduction

Following each installation of a Varian ***MERCURY*** NMR spectrometer system, an installation engineer tests and demonstrates the instrument's operation.

1.1 Overview

The procedures for the acceptance tests, as well as specifications, are provided in this manual. The forms for entering the test results are provided in Chapter 5, "Acceptance Test Results," beginning on page 75. The forms follow the same sequence as the tests.

Acceptance Tests

The objectives of the acceptance tests procedures are threefold:

- To identify the tests to be performed during system installation.
- To identify the precise methods by which these tests are performed.
- To leave the instrument in a calibrated, ready to use, state.

The procedures are arranged by the type of specification. The arrangement of the procedures does not matter (although some procedures use results from other procedures) and is determined by the installation engineer. These procedures cover the basic specifications of the instrument—signal-to-noise (S/N), resolution, and lineshape—and are not intended to reflect the full range of operating capabilities or features of a ***MERCURY*** NMR spectrometer. *Performance of any additional tests beyond those described in this manual must be agreed upon in writing as part of the customer contract.*

Acceptance Specifications

All specifications are subject to change without notice. The specifications published in this manual shall prevail unless negotiation or customer contract determines otherwise. Refer to the text in each chapter for other conditions. *Request of any additional specifications beyond those listed in this manual must be agreed upon in writing as part of the customer contract.*

The following policies are in effect at installation:

- *Specifications Policy for Probes Used in Systems other than* ***MERCURY***—No guarantee is given that probes purchased for use in systems other than ***MERCURY*** will meet current specifications.
- *Testing Policy for Indirect Detection Probes used for Direct Observe Broadband Performance*—Probes designed for indirect detection applications are tested for

indirect detection performance only. Indirect detection acceptance tests are performed only if an indirect detection probe was purchased for use with the ***MERCURY***.

- *Sample Tubes Policy*—Tests are performed in 5-mm sample tubes with 0.38 mm wall thickness (Wilmad 528-PP, or equivalent) and 10-mm sample tubes with 0.46 mm wall thickness (Wilmad 513-7PP, or equivalent). Using sample tubes with thinner walls (Wilmad 5-mm 545-PPT, or equivalent; Wilmad 10-mm 513-7PPT, or equivalent) increases signal-to-noise.

Computer Audit

A computer audit form is included in "Computer Audit" on page 77. The information from this form will help Varian assist you better in distributing future software upgrades and avoiding hardware compatibility problems. You are asked for information about all computers directly connected to the spectrometer or else used to process NMR data.

Installation Checklist

An installation checklist form is given in "System Installation Checklist" on page 79.

System Documentation Review

Following the completion of the acceptance tests and computer audit, the installation engineer will review the following system documentation with the customer:

- Software Object Code License Agreement.
- Varian and OEM manuals.
- Warranty coverage and where to telephone for information.

Basic System Demonstration

The installation engineer will also demonstrate the basic operation of the system to the laboratory staff. The objective of the demonstration is to familiarize the customer with system features and safety requirements, as well as to assure that all mechanical and electrical functions are operating properly.

The system demonstration includes the following items:

Magnet Demonstration

- Posting requirements for magnetic field warning signs
- Cryogenics handling procedures and safety precautions
- Magnet refilling
- Flowmeters
- Homogeneity disturbances

Console and Probe Demonstration

- Loading programs.

- Experiment setup, including installing the probe in the magnet.
- Basic instrument operation to obtain typical spectra, including probe tuning, magnet homogeneity shimming, and printer/plotter operation. (*Note that Varian installation engineers are neither responsible for nor trained to run any spectra not described in the Acceptance Tests Procedures.*)
- Demonstration of *GLIDE* ^{1}H and ^{13}C operation.
- Demonstration of *GLIDE* APT and DEPT experiments.
- Demonstration of (optional) homonuclear decoupling.

Detailed specifications and circuit descriptions are not covered.

Formal training in the operation and maintenance of the spectrometer is conducted by Varian at periodically scheduled training seminars held in most Varian Application Laboratories. On-site training is available in some geographic locations. Contact your sales representative for further information on availability and pricing for these courses.

To make the system demonstration most beneficial, the customer should review Varian and OEM operation and reference manuals before viewing the demonstration.

1.2 General Acceptance Testing Requirements

Each Varian ***MERCURY*** spectrometer is designed to provide high-resolution performance when operated in an environment as specified in the ***MERCURY*** *Installation Planning Guide*. Unless both the specific requirements of this *Acceptance Test Procedures* manual and the general requirements specified in the ***MERCURY*** *Installation Planning Guide* are met, Varian cannot warrant that the NMR spectrometer system will meet the published specifications.

General Requirements

- The ***MERCURY*** performance specifications in effect at the time of your order are used to evaluate the system.
- The appropriate quarter-wavelength cable must be used for each nucleus.
- Homogeneity settings must be optimized for each sample (manual shimming may be required in any or all cases). The shim parameters for resolution tests on each probe should be recorded in a log book and in a separate file name (in the directory `/vnmr/shims`) for each probe. For example, for a 5-mm switchable probe, the shim parameters can be saved with the command `svs('sw5res')`. These values can then be used as a starting point when adjusting the homogeneity on unknown samples, by the command `rts('sw5res')`.
- The probe must be tuned to the appropriate frequency.
- The spinning speed must be set to the following:

Sample (mm)	*Nuclei*	*Speed (Hz)*
5	all	20–26
10	all	15

Spinning 10-mm tubes faster than 15 Hz may cause vortexing in samples, severely degrading the resolution.

- Some test parameters are stored in the disk library `/vnmr/tests` and can be recalled by entering `rtp('/vnmr/tests/xxx')`, where *xxx* is the name of the file that contains the parameters to be retrieved—for example, `rtp('/vnmr/tests/H1sn')`. To see the parameter sets available for the standard tests, enter `ls('/vnmr/tests')`. Other sets come from `/vnmr/stdpar`.
- For all sensitivity tests, the value of `pw` must be changed to the value of the 90° pulse found in the pulse width test on the same probe.
- During calibration, *GLIDE* creates an appropriate `pw` array to determine the 90° pulse width. For manually run observe pulse width tests, an appropriate array of `pw` values must be entered to determine the 180° pulse. The 180° pulse is the first non-zero pulse that gives minimum intensity of the spectrum. The 180° pulse is usually determined by interpolation between a value that gives a positive signal, and a value that gives a negative signal. The 90° pulse width is one half the 180° pulse.
- Signal-to-noise (S/N) is measured by the computer as follows:

$$S/N = \frac{\text{maximum amplitude of peak}}{2 \times \text{root mean square of noise region}}$$

- Lineshape should be measured digitally with the aid of the system software. The properly scaled spectra should also be plotted and the plot retained.
- Digital determination of lineshape:
  1. Display and expand the desired peak.
  2. Enter `nm`, then `dc` for drift correction to ensure a flat baseline. Set `vs=10000`. Click the menu button labeled `Th` to display the horizontal threshold cursor. Set `th=55` (the 0.55% level).
  3. Click the menu button labeled Cursor or Box until two vertical cursors are displayed, and align them on the intersections of the horizontal cursor and the peak. Type `delta?` to see the difference in Hz between the cursors.
  4. Set `th=11` (the 0.11% level) and repeat.
- Determination of lineshape from a plot:
  1. Use a large enough plot width to allow accurate determination of the baseline. The baseline should be drawn through the center of the noise, in a region of the spectrum with no peaks.
  2. The 0.55% and 0.11% levels are then measured from the baseline and calculated from the height of the peak and the value of `vs`. For example, if a peak is 9.0 cm high with `vs=200`, then the 0.55% level on a 100-fold vertical expansion (`vs=20000`) is 9.0×0.55, or 4.95 cm from the baseline.

  If the noise is significant at the 0.55% and 0.11% levels, the linewidth should be measured horizontally to the center of the noise.
- For all sensitivity tests, a noise region free of any anomalous features should be chosen with the cursors. Neither cursor should be placed any closer to an edge of the spectrum than 10 percent of the value of `sw`. This should produce the best possible signal-to-noise that is representative of the spectrum.
- The results of all tests should be plotted to create a permanent record. Include a descriptive label and a list of parameters. These plots can then be saved as part of the acceptance tests documentation.

Part 1:

Acceptance Test Procedures

Chapter 2 **Liquids Probes Test Procedures**

Chapter 3 **Console and Magnet Test Procedures**

Chapter 2. Liquids Probes Test Procedures

This chapter contains procedures for both required and optional liquids probes tests. Specifications to be demonstrated are contained in Part 2 of this manual.

Write the results of each probe test on the forms provided in “Liquids Probes Test Results” on page 83.

The following is a list of the sections in this chapter:

- 2.1 “How to Test a Probe,” page 18
- 2.2 “Probe Calibration Files,” page 19
- 2.3 “Resolution, Lineshape, and Spinning Sidebands Procedures,” page 20
- 2.4 “90 Pulse Width Procedures,” page 25
- 2.5 “Sensitivity Procedures,” page 42
- 2.6 “Configuring Solvent-Based Shims and setlk for GLIDE,” page 51

2.1 How to Test a Probe

This section provides the basic steps for testing a probe.

1. Create a probe calibration file as described in "Probe Calibration Files" on page 19.
2. Follow the appropriate test procedures listed in Table 1 and Table 2 for the probe. If you wish, you can check off each test as it is finished.

   Table 1 lists the tests for probes that observe ^{1}H, ^{13}C, ^{19}F, or ^{13}P. Table 2 lists the test for probes that observe ^{15}N or ^{29}Si and probes capable of indirect detection.
3. After the appropriate tests are completed for the probe, print the corresponding probe calibration file by entering the following command in the VNMR input window:

   `ACreport`
4. Follow the instructions Section 2.6 for configuring solvent-based shims and the `setlk` macro for proper *GLIDE* and automation operation.

Table 1. Test order for each probe that observes ^{1}H, ^{13}C, ^{19}F, or ^{31}P

Done	*Test*
_____	1H Spinning Resolution and Lineshape (50%, 0.55%, 0.11%) of CHCl3, on page 21
_____	1H Spinning Sidebands of CHCl3, on page 23
_____	1H Observe 90 Pulse Width, on page 26
_____	1H Sensitivity, on page 43
_____	13C Resolution Test, on page 24
_____	13C Observe 90 Pulse Width and gH2, on page 29
_____	13C Sensitivity, on page 47
_____	19F Observe 90 Pulse Width, on page 27
_____	19F Sensitivity, on page 45
_____	31P Observe 90 Pulse Width, on page 28
_____	31P Sensitivity, on page 46

Table 2. Test order for probes that observe ^{29}Si, ^{15}N, or indirect detection

Done	*Test*
_____	29Si Observe 90 Pulse Width (Only 4-Nucleus Probes with 29Si), on page 30
_____	29Si Sensitivity (Only 4-Nucleus Probes with 29Si), on page 49
_____	15N Observe 90 Pulse Width (Only 400-MHz or 4-Nuc Probes with 15N), on page 31
_____	15N Sensitivity (Only 400-MHz or 4-Nucleus Probes with 15N), on page 50
_____	13C pwx90 Pulse Width, on page 32
_____	31P pwx90 Pulse Width, on page 36
_____	15N pwx90 Pulse Width (Only 400-MHz with Indirect Detection Probes), on page 39

2.2 Probe Calibration Files

VNMR 5.3 software includes probe calibration files in the directory `/vnmr/probes`. Probe calibration files contain `pw90`, `tpwr`, `dmf`, `dpwr`, and `pwx` settings for each probe used with the system. *GLIDE*, automation, and manual operation using macros—including `h1`, `hcosy`, and `dept`—retrieve appropriate power levels, the `pw90` value, and the `dmf` value from the probe calibration file, instead of using preset values in the parameters files. **Successful operation depends on the existence of a probe calibration file for each probe that is used with the system.**

When the ***MERCURY*** NMR spectrometer is installed, the proper probe calibration files are set up, so that the system is ready to use when the installer leaves the site.

The probe calibration files must be created when the system is installed or the first time a probe is used. Create or change the probe calibration file as follows:

To Create a Probe Calibration File

Do these steps for each probe that is tested.

1. Log in as `vnmr1`.
2. Enter the following command in the VNMR input window:

   `addprobe('probe_name','system')`

   Where *`probe_name`* is a descriptive name for the probe. For example, `sw5mm` for a 5-mm switchable probe, or `4nucPFG` for a 4-nucleus PFG probe. The `'system'` argument makes the probe calibration files for this probe avaible to all users by placing the files in `/vnmr/probes`. Without the argument, the files are placed in the `vnmrsys` directory of the user who entered the command (e.g., `~vnmr1/vnmrsys/probes`) and are only available to that user..

   The values for power, `pw90`, `dmf`, and `pp` are initially set to 0 and filled in later by running the calibration procedures for ^{1}H, ^{19}F, ^{31}P, and ^{13}C using *GLIDE*.

To Change to a Different Probe Calibration File

After the probes are tested and calibrated, use this procedure to switch to the appropriate calibration file when a different probe is installed.

1. Log in as `vnmr1`.
2. Enter the following command in the VNMR input window;

   `probe='probe_name'`

   Where *`probe_name`* is the name of the directory in `/vnmr/probes` named for the new probe (e.g., `sw5mm`).

2.3 Resolution, Lineshape, and Spinning Sidebands Procedures

The procedures in this section demonstrate the resolution, lineshape, and spinning sidebands (SSB) specifications listed in “Resolution, Lineshape, Spinning Sidebands Specifications” on page 62.

- 1H Spinning Resolution and Lineshape (50%, 0.55%, 0.11%) of CHCl3
- 1H Spinning Sidebands of CHCl3
- 13C Resolution Test

^{1}H Spinning Resolution and Lineshape (50%, 0.55%, 0.11%) of $CHCl_3$

Samples

Amplitude Level	*Sample Tube (mm)*	*Test Sample*	*Part No.*
50.0%, 0.55%, 0.11%	5	20% chloroform in acetone–d_6	00-968120-76
50.0%, 0.55%, 0.11%	5	5% chloroform in acetone–d_6	00-968120-99
50.0%, 0.55%, 0.11%	4	5% chloroform in acetone–d_6	00-993143-99
50.0%, 0.55%, 0.11%	5	1% chloroform in acetone–d_6	00-968120-89
50.0%, 0.55%, 0.11%	10	1% chloroform in acetone–d_6	00-968123-89

Procedure

The ^{1}H lineshape test (this test) and the ^{1}H spinning sidebands test (the next test) *must be passed simultaneously* and both tests plotted together.

1. Insert the appropriate $CHCl_3$ sample in the magnet and spin it.
2. Enter `rtp('/vnmr/tests/H1lshp') nm` and set `nt=1 vs=100`. Enter `su` to set up the system hardware.
3. Tune the probe.
4. Enter a value for `pw` appropriate for your sample:

If your sample is:	*Then use the value for a:*
1% $CHCl_3$, (00-968120-89)	90 pulse
5% $CHCl_3$, (00-968120-99)	30 pulse
20% $CHCl_3$, (00-968120-76)	20 pulse

5. Enter `ga` to acquire the spectrum. Phase the spectrum, set `wp=250`, and plot using `pl`. Increase `vs` by a factor of 100 times and plot the expanded spectrum using `pl pscale page`.

   If floor vibration results in excessive noise around the base of the peak, `nt` can be set to a larger value (e.g., `nt=4` or `nt=16`); however, if extreme vibrations are present, it may be impossible to measure the lineshape accurately.

6. Measure lineshape as the linewidth of the $CHCl_3$ peak at 50%, 0.55% and 0.11% of the main peak amplitude. Refer to Figure 1 and use the substeps below to determine the lineshape:
   a. Display and expand the desired peak.
   b. Enter `nm`, then `dc` for drift correction to ensure a flat baseline. Set `vs=10000`. Place a cursor on the chloroform line and enter `nl dres` (the 50% level).
   c. Enter `nm`, then `dc` for drift correction to ensure a flat baseline. Set `vs=10000`. Click the VNMR menu button labeled `Th` to display the horizontal threshold cursor. Set `th=55` (the 0.55% level).

d. Click the first menu button labeled Cursor or Box to display two vertical cursors, and align them on the intersections of the horizontal cursor and the peak. Enter `delta?` to see the difference in Hz between the cursors.

e. Set `th=11` (the 0.11% level) and repeat.

7. Write the results for each probe in the forms provided in "Liquids Probes Test Results" on page 83.

8. After the specifications are met, do the following:

   - Write down the values for `z0`, `lockpower`, and `lockgain` as they appear in the `Acqi` window. Use the forms provided in "Liquids Probes Test Results" on page 83.
   - Save the shims by entering the following command in the VNMR input window:

     ```
     svs('acetone')
     ```

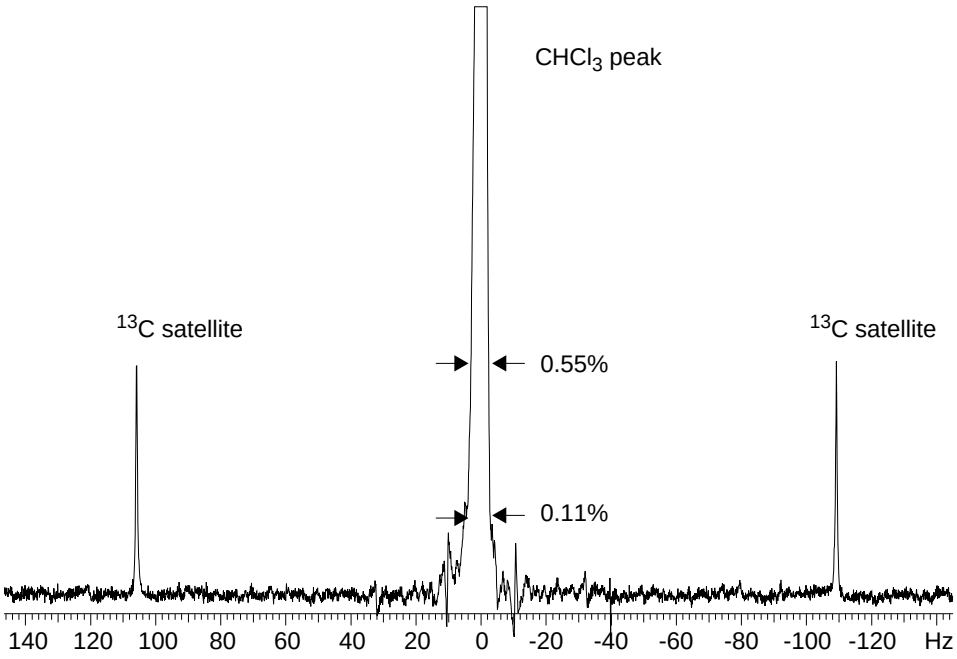

Figure 1. ^{1}H lineshape spinning measurement

^{1}H Spinning Sidebands of $CHCl_3$

Samples

Amplitude Level	*Sample Tube (mm)*	*Test Sample*	*Part No.*
50.0%, 0.55%, 0.11%	5	20% chloroform in acetone–d_6	00-968120-76
50.0%, 0.55%, 0.11%	5	5% chloroform in acetone–d_6	00-968120-99
50.0%, 0.55%, 0.11%	4	5% chloroform in acetone–d_6	00-993143-99
50.0%, 0.55%, 0.11%	5	1% chloroform in acetone–d_6	00-968120-89
50.0%, 0.55%, 0.11%	10	1% chloroform in acetone–d_6	00-968123-89

Procedure

The ^{1}H spinning sidebands test (this test) and the ^{1}H lineshape test (the previous test) *must be passed simultaneously* and both tests plotted together.

1. Using the appropriate $CHCl_3$ sample, measure the ^{1}H spinning sidebands on the same spectrum as the ^{1}H lineshape test.
2. Use the spectra and parameter set from the lineshape test. Plot the spectrum again using a large enough value of `wp` to show all the spinning sidebands.
3. Measure spinning sideband amplitudes as a percentage of the main peak.

   Spinning sidebands occur at frequency intervals on either side of the central peak equal to the spinning rate. The sidebands might not be split.
4. The standard test requires `nt=4`. If the sidebands meet specifications at `nt=4`, repeating the test at `nt=16` is not necessary.
5. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

^{13}C Resolution Test

Samples

Amplitude Level	*Sample Tube (mm)*	*Test Sample*	*Part No.*
50.0%, 0.55%, 0.11%	5	40% p-dioxane in benzene–d_6 (ASTM)	00-968120-69
50.0%, 0.55%, 0.11%	10	40% p-dioxane in benzene–d_6 (ASTM)	00-968123-69

Procedure

1. Enter `rtp('/vnmr/tests/C13res') su`.
2. Tune the probe.
3. Set `nt=4` and `lb='n'`. Set the decoupler modulation to `dmm='c'` and `d1=60`.
4. Set the sample spinning rate at 20 ± 5 Hz.
5. Enter `ga` to acquire the spectrum. Plot the spectrum using `wp=50`, and use `dres` to determine the linewidth at 50% of the decoupled peak.
6. If decoupling is not complete, run a proton spectrum and set the cursor at the center of the single peak. Enter `movetof`, then enter `tof?` to display the new values of `tof`. Record the value.
7. Enter `jexp`*n*, where *n* is the experiment number of the carbon experiment (e.g., `jexp2` to join experiment 2), and set `dof` to the value of `tof` obtained in the proton spectrum.
8. Repeat step 5.
9. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

2.4 90 Pulse Width Procedures

This section contains the procedures required to demonstrate the specifications listed in “90 Pulse Width and gH2 Specifications” on page 65.

- 1H Observe 90 Pulse Width
- 19F Observe 90 Pulse Width
- 31P Observe 90 Pulse Width
- 13C Observe 90 Pulse Width and gH2
- 29Si Observe 90 Pulse Width (Only 4-Nucleus Probes with 29Si)
- 15N Observe 90 Pulse Width (Only 400-MHz or 4-Nuc Probes with 15N)
- 13C pwx90 Pulse Width
- 31P pwx90 Pulse Width
- 15N pwx90 Pulse Width (Only 400-MHz with Indirect Detection Probes)

Refer to other Varian manuals for further information on pulse width determination.

^{1}H Observe 90 Pulse Width

Samples

Sample Tube (mm)	*Test Sample*	*Sample Part Number*
5	0.1% ethylbenzene, 0.01% TMS 99.89% deuterochloroform ($CDCl_3$)	00-968120-70
10	0.1% ethylbenzene, 0.01% TMS 99.89% deuterochloroform ($CDCl_3$)	00-968123-70
4	0.1% ethylbenzene, 0.01% TMS 99.89% deuterochloroform ($CDCl_3$)	00-993143-99

Procedure

This procedure calibrates `tpwr` and the 90 pulse width for ^{1}H.

1. Open *GLIDE* by clicking the GLIDE button in the VNMR menu.
2. Click on the Setup icon.
3. Insert the sample in the magnet using either the manual button on the magnet leg or the Insert button in the Setup window.
4. Select Calibrate Proton from the Experiment drop-down menu. You do not need to select a solvent.
5. Click the Setup button at the bottom of the Setup window.
6. Tune the probe.
7. Click on the Acquire button under Custom.
8. If you have already locked and shimmed the sample, turn off autolock and autoshim.
9. Enter an appropriate value for `pwmax`, which is listed in Table 10 and Table 11 on page 66.
10. Click on Close at the bottom of the Acquire window.
11. Click the Go icon in the *GLIDE* window.

    You should get a plot of arrayed spectra.
12. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

^{19}F Observe 90 Pulse Width

Sample

Sample Tube (mm)	*Test Sample*	*Sample Part Number*
5	0.05% trifluorotoluene in benzene–d_6	00-968120-82

Procedure

This procedure calibrates `tpwr` and the 90 pulse width for ^{19}F.

1. Open *GLIDE* by clicking the GLIDE button in the VNMR menu.
2. Click on the Setup icon.
3. Insert the sample in the magnet using either the manual button on the magnet leg or the Insert button in the Setup window.
4. Select Calibrate Florine from the Experiment drop-down menu. You do not need to select a solvent.
5. Click the Setup button at the bottom of the Setup window.
6. Tune the probe.
7. Click on the Acquire button under Custom.
8. If you have already locked and shimmed the sample, turn off autolock and autoshim.
9. Enter an appropriate value for `pwmax`, which is listed in Table 10 and Table 11 on page 66.
10. Click on Close at the bottom of the Acquire window.
11. Click the Go icon in the *GLIDE* window.

    You should get a plot of arrayed spectra.
12. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

^{31}P Observe 90 Pulse Width

Samples

Sample Tube (mm)	*Test Sample*	*Sample Part Number*
5	0.0485 M triphenylphosphate in $CDCl_3$	00-968120-87
10	0.0485 M triphenylphosphate in $CDCl_3$	00-968123-87

Procedure

This procedure calibrates `tpwr` and the 90 pulse width for ^{31}P.

1. Make sure the appropriate quarter-wavelength cable is installed.
2. Open *GLIDE* by clicking the GLIDE button in the VNMR menu.
3. Click on the Setup icon.
4. Insert the sample in the magnet using either the manual button on the magnet leg or the Insert button in the Setup window.
5. Select Calibrate Phosphorus from the Experiment drop-down menu. You do not need to select a solvent.
6. Click the Setup button at the bottom of the Setup window.
7. Tune the probe.
8. Click on the Acquire button under Custom.
9. If you have already locked and shimmed the sample, turn off autolock and autoshim.
10. Enter an appropriate value for `pwmax`, which is listed in Table 10 and Table 11 on page 66.
11. Click on Close at the bottom of the Acquire window.
12. Click the Go icon in the *GLIDE* window.

    You should get a plot of arrayed spectra.
13. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

^{13}C Observe 90 Pulse Width and γH_2

Samples

Sample Tube (mm)	*Test Sample*	*Sample Part Number*
5	40% p-dioxane in benzene–d_6 (ASTM)	00-968120-69
10	40% p-dioxane in benzene–d_6 (ASTM)	00-968123-69

Procedure

This procedure calibrates `tpwr`, the 90 pulse width, `dpwr`, and γH_2 for ^{13}C, as well set `dmf`, `pplvl`, and `pp`.

1. Make sure the appropriate quarter-wavelength cable is installed.
2. Open *GLIDE* by clicking the GLIDE button in the VNMR menu.
3. Click on the Setup button.
4. Insert the sample in the magnet using either the manual button the magnet leg or the Insert button in the Setup window.
5. Select Calibrate Carbon from the Experiment drop-down menu. You do not need to select a solvent.
6. Click the Setup button at the bottom of the Setup window.
7. Tune the probe.
8. Click on the Acquire button under Custom.
9. If you have already locked and shimmed the sample, turn off autolock and autoshim.
10. Enter an appropriate value for `pwmax`, which is listed in Table 10 and Table 11 on page 66.
11. Click on Close at the bottom of the Acquire window.
12. Click the Go icon in the *GLIDE* window.

    You should get a plot of arrayed spectra for 90 pulse width and γH_2, as well as arrayed spectra for `pp`.
13. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

^{29}Si Observe 90 Pulse Width (Only 4-Nucleus Probes with ^{29}Si)

This test is only for 4-nucleus probes with ^{29}Si.

Sample

Sample Tube (mm)	*Test Sample*	*Sample Part Number*
5	25% hexamethyldisiloxane in benzene–d_6	00-968120-98

Procedure

1. Make sure the appropriate quarter-wavelength cable is installed.
2. Insert the sample into the magnet.
3. Enter `rtp('/vnmr/tests/Si29sn') su`.
4. Tune the probe observe channel without using the filter.
5. Enter `addnucleus('Si29')` to add an entry for ^{29}Si to the probe calibration file.
6. Set `d1=200 at=6 fn=8k sw=2k dm='y' dmm='w'`
7. If you already calibrated ^{13}C, you can retrieve `dpwr` and `dmf` with the following commands:

   ```
   getparam('dpwr','H1'):dpwr
   getparam('dmf','H1'):dmf
   ```

8. Enter `ga`.
9. Enter `ds`. Set `vs=50` and adjust `vs` so that the peak occupies about half the screen.
10. Set a cursor on the peak. Enter `movetof pw=10,20,30,40,50,60,70,80 ga`.
11. When the last spectrum is obtained, enter `dssh`.

    The first null spectra is the 180 pulse; therefore, the 90 pulse width is one half the 180 pulse.

12. Store the `tpwr` used and the 90 pulse width found by entering the following commands in the VNMR input window:

    ```
    setparams('tpwr','58')
    setparams('pw90','12.5')
    ```

    The values are treated as strings; therefore, they are enclosed in single quotes.

13. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

^{15}N Observe 90 Pulse Width (Only 400-MHz or 4-Nuc Probes with ^{15}N)

This test is performed at installation on only 400-MHz systems and broadband systems with 5-mm ^{1}H/^{19}F/^{13}C/^{15}N 4-nucleus probes, unless explicitly agreed upon in writing as part of the customer contract.

Samples

Sample Tube (mm)	*Test Sample*	*Sample Part Number*
5	90% formamide in DMSO–d_6	00-968120-83
10	90% formamide in DMSO–d_6	00-968123-83

Procedure

1. Make sure the appropriate quarter-wavelength cable and probe tuning rod are installed.
2. Insert the sample into the magnet.
3. Enter `rtp('/vnmr/tests/N15sn') su`.
4. Tune the probe observe channel.
5. Set `d1=200 at=6 fn=8k sw=2k dm='y' dmm='w'`
6. If you already calibrated ^{13}C, you can retrieve `dpwr` and `dmf` with the following commands:

   ```
   getparam('dpwr','H1'):dpwr
   getparam('dmf','H1'):dmf
   ```

7. Enter `ga`.
8. Enter `ds`. Set `vs=50` and adjust `vs` so that the peak occupies about half the screen.
9. Set a cursor on the peak. Enter `movetof pw=10,20,30,40,50,60,70,80 ga`.
10. When the last spectrum is obtained, enter `dssh`.

    The first null spectra is the 180 pulse; therefore, the 90 pulse width is one half the 180 pulse.

11. Store the `tpwr` used and the 90 pulse width found by entering the following commands in the VNMR input window:

    ```
    setparams('tpwr','58')
    setparams('pw90','12.5')
    ```

    The values are treated as strings; therefore, they are enclosed in single quotes.

12. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

^{13}C pwx90 Pulse Width

This test is only for systems with indirect detection probes.

Sample

Sample Tube (mm)	*Test Sample*	*Sample Part Number*
5	1% iodomethane–^{13}C, 1% trimethylphosphite, 0.2% Cr(acac) in $CDCl_3$	00-968120-96

Procedure

To perform the ^{13}C or X-nucleus 90 pulse with calibration in the indirect mode, use the HMQC experiment.

1. Make sure the appropriate quarter-wavelength cable is installed.
2. Insert the 1% iodomethane–^{13}C sample (Part No. 00-968120-96). Tune the probe on this sample.
3. Enter `rtp('/vnmr/tests/H1sn')` to retrieve the parameters from the ^{1}H sensitivity measurement.
4. Set `pw` and `tpwr` to the values determined for the ^{1}H sensitivity procedure; you can get these values from the probe calibration file by entering:

   ```
   getparam('tpwr'):tpwr
   getparam('pw90'):pw
   ```

5. Set `gain=10 dn='C13' d1=5` and enter `ga`.
6. Place two cursors around the region with the peaks. Enter `movesw` to narrow the spectral width.
7. Determine the 90 pulse width by arraying`pw` to ≥360 pulse width. Enter the macro `array` to set up the array. As the macro displays the following prompts, type in the response shown in bold:

   ```
   parameter to be arrayed:          pw
   number of steps in the array:     25
   starting increment:               2
   array increment:                  2
   ```

   In the above example, the experiment is set up to array the `pw` parameter from 2 µs to 52 µs, in 2 µs increments. If the 90 pulse width is 10 µs, the 450 pulse width corresponds to `pw=50` µs (5 * `pw90`).
8. Enter `ga` to start the acquisition. After the last spectrum is finished, enter `ds(5) vp=50` to display the fifth spectrum. Enter `aph` to phase the spectrum, then enter `ai dssh` to display the arrayed spectra.
9. Set `pw` equal to the 90 pulse width you have determined. Ether `ga` to acquire the spectrum. Enter `f ds` to display the spectrum.

   The spectrum should appear similar to the example shown in Figure 2. The large, three-line pattern at about 2.2 ppm is the signal of interest.

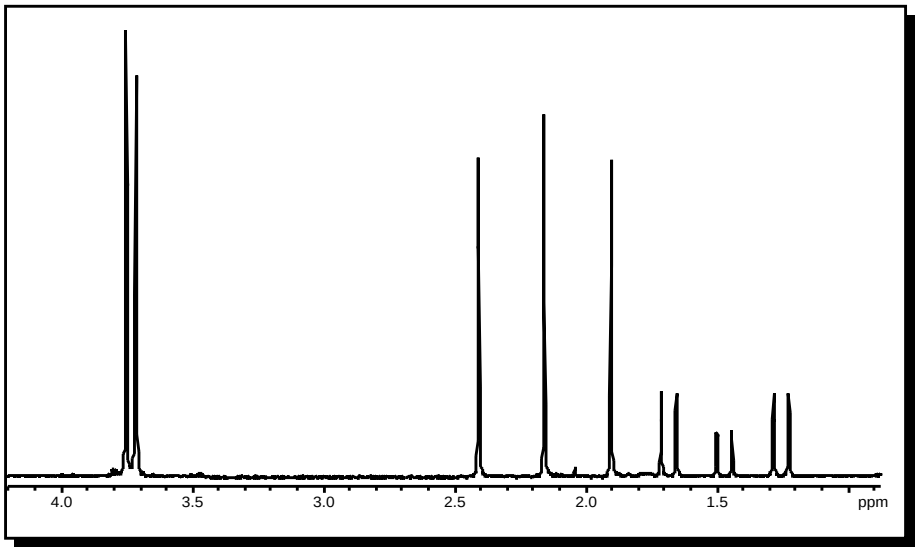

Figure 2. Normal ^{1}H spectrum of $^{13}CH_3I$

10. To perform the ^{13}C X-nucleus 90 pulse width calibration in the indirect mode using HMQC, enter `hmqc` in the same experiment or move the parameters to a different experiment by entering `mp(x,y)`, where `x` is the current experiment and `y` is the experiment to which the parameter set is to be moved.

11. Set the following parameters: `fn=8192`, `ni=1`, `phase=1`, `nt=1`, `ss=0`, `spin=0` (use `acqi` to turn the spinner off if the optional spinner hardware is not installed), `d1=5`, `pw` to the ^{1}H 90 pulse width(determined from the previous steps), `tpwr` to the `tpwr` level to give the ^{1}H 90 pulse width (determined from the previous steps), `null=0`, `j=151`, `tof` to the `tof` determined from the ^{1}H spectrum of the 1% iodomethane-^{13}C in step 6 above, `dn='C13'`, `dm='nnn'`, and `dof` from the following table:

^{1}H freq. (MHz)	*^{13}C (Hz)*	*^{15}N (Hz)*	*^{31}P (Hz)*
400	–965	–12000	9000
300	–9000	–9200	7000

12. To determine the ^{13}C X-nucleus 90 pulse width and rf homogeneity using the HMQC pulse sequence, `pwx` is arrayed for a particular `pwxlvl`. The spectrum corresponding to the ^{13}C X-nucleus 90 pulse width using the HMQC pulse sequence is the maximum amplitude spectrum.

    An array of spectra appears as shown in Figure 3. The array shows only the response of the outer line (at about 2.4 ppm) of the three-line pattern. In HMQC, `null` occurs at 45, 135 maximum at 90 and negative at 180 .

    Set `pwxlvl` to the `tpwr` value for normal ^{13}C observe (to get this value you can enter `getparam('tpwr','C13'):pwxlvl`). Enter `array` to array `pwx`. As `array` displays the following prompts, enter in the responses shown in bold:

```
parameter to be arrayed:          pwx
number of steps in the array:     20
starting increment:               2
array increment:                  1
```

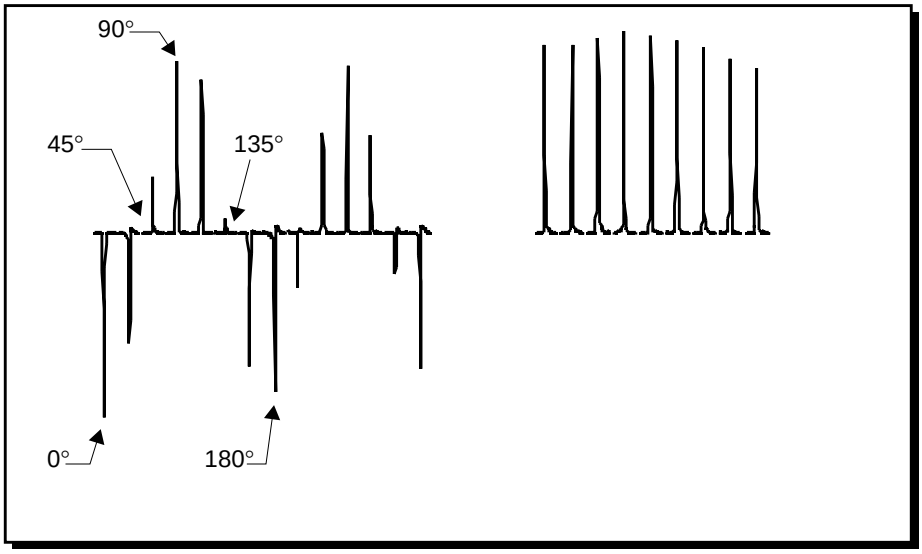

Figure 3. pwx calibration, coarse (left) and fine (right)

In this example, the experiment is set up to array pw from 2 µs to 22 µs, in 1 µs increments.

13. Enter ga to start the acquisition. After the last spectrum is finished, enter ds(1) to display the first spectrum. Enter aph to phase the spectrum, then enter ai dssh to display the arrayed spectra.
14. Determine the pwx90 from the first maximum. Write the results for each probe in the forms provided in "Liquids Probes Test Results" on page 83.
15. To store the values in the probe calibrations file, enter the following commands:

    ```
    setparams('pwxlvl','61.0','C13')
    setparams('pwx','14.2','C13')
    ```

    Be sure to enclose the values in single quotes because they are treated as strings.

^{13}C Decoupling Calibration—Measuring γH_2 for Indirect Detection

This procedure describes how to perform ^{13}C decoupling calibration as well as how to measure γH_2 for indirect detection.

1. Set pwx equal to the value determined in step 14 in the previous procedure.
2. Enter ga to acquire a spectrum.

   The spectrum should look like the second spectrum in Figure 4, with the outer two peaks of the three line pattern up and the center peak down.
3. Set dmm='ccc' dm='nny' dpwr=30 dof=dof+2000,dof–2000. Then enter ga.
4. Enter ds(1), place a cursor on each of the positive peaks, and write down the delta.
5. Enter ds(2), place a cursor on each of the positive peaks, and write down the delta.
6. Enter h2cal and enter the delta values for the high field and low field coupling.

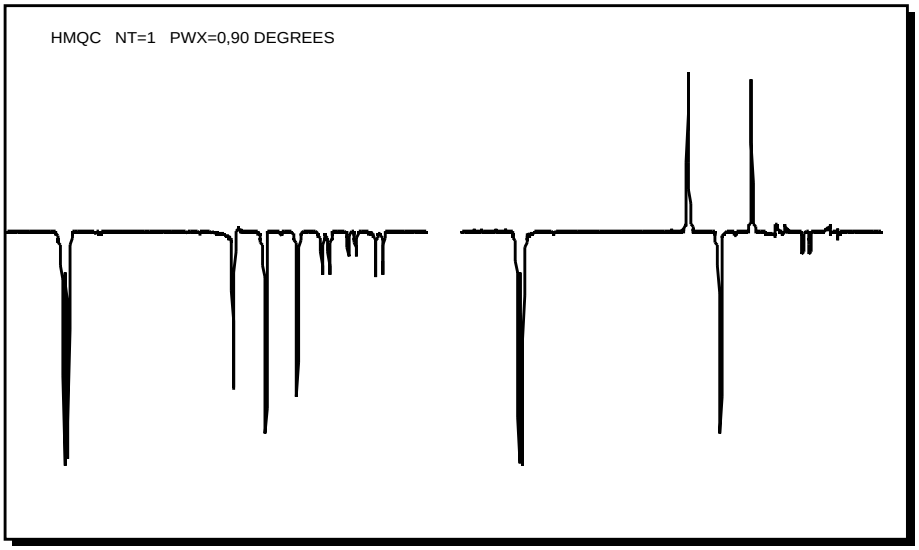

Figure 4. HMQC with and without X-nucleus pulses

7. When prompted, enter `151` for the coupling constant.

   A γH_2 of 5000 is necessary for decoupling in indirect detection. If the value is not 5000, increase `dpwr` by 3 dB until γH_2 is 5000.

8. To store the values in the probe calibrations file, enter the following commands:

   ```
   setparams('dpwr','43','C13')
   setparams('dmf','20000','C13')
   ```

   Be sure to enclose the values in single quotes because they are treated as strings.

^{31}P pwx90 Pulse Width

This test is only for systems with indirect detection probes.

Sample

Sample Tube (mm)	*Test Sample*	*Sample Part Number*
5	1% iodomethane–^{13}C, 1% trimethylphosphite, 0.2% Cr(acac) in $CDCl_3$	00-968120-96

Procedure

To perform the ^{31}P or X-nucleus 90 pulse width calibration in the indirect mode, use the HMQC pulse sequence.

1. Make sure the appropriate quarter-wavelength cable is installed.
2. Insert the sample. Tune the probe on this sample.
3. Enter `rtp('/vnmr/tests/H1sn')` to retrieve the parameters from the ^{1}H sensitivity measurement.
4. Set `pw` and `tpwr` to the values determined for the ^{1}H sensitivity procedure; you can get these values from the probe calibration file by entering:

   ```
   getparam('tpwr'):tpwr
   getparam('pw90'):pw
   ```

5. Set `gain=10 dn='P31' d1=5` and enter `ga`.
6. Place two cursors around the region with the peaks. Enter `movesw` to narrow the spectral width.
7. Determine the 90 pulse width by arraying`pw` to ≥360 pulse width. Enter the macro `array` to set up the array. As the macro displays the prompts, type in the response shown in bold:

   ```
   parameter to be arrayed:          pw
   number of steps in the array:     25
   starting increment:               2
   array increment:                  2
   ```

   In the above example, the experiment is set up to array `pw` from 2 µs to 52 µs, in 2 µs increments. If the 90 pulse width is 10 µs, the 450 pulse width corresponds to `pw=50` µs (5 * `pw90`).
8. Enter `ga` to start the acquisition. After the last spectrum is finished, enter `ds(5) vp=50` to display the fifth spectrum. Enter `aph` to phase the spectrum; then enter `ai dssh` to display the arrayed spectra.
9. Set `pw` equal to the 90 pulse width you have determined. Enter `ga` to acquire the spectrum. Enter `f ds` to display the spectrum.

   The spectrum should appear similar to the example shown in Figure 5. The large, two-line pattern at about 3.75 ppm is the signal of interest.
10. To perform the ^{31}P X-nucleus 90 pulse width calibration in the indirect mode using HMQC, enter `hmqc` in the same experiment or move the parameters to a

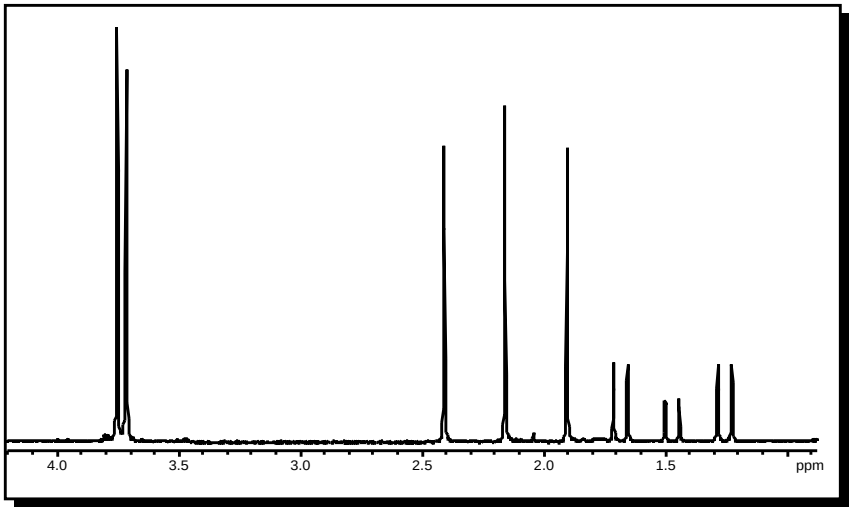

Figure 5. Normal ^{1}H spectrum of $^{13}CH_3I$

different experiment by entering `mp(x,y)`, where `x` is the current experiment and `y` is the experiment to which the parameter set is to be moved.

11. Set the following parameters: `fn=8192 ni=1 phase=1 nt=1 ss=0 spin=0` (use `acqi` to turn the spinner off if the optional spinner hardware is not installed) `d1=2 pw` to the ^{1}H 90 pulse width(determined from the previous steps) `tpwr` to the `tpwr` level to give the ^{1}H 90 pulse width (determined from the previous steps), `null=0`, `j=10.5`, `tof` to the `tof` determined from the ^{1}H spectrum of the 1% iodomethane-^{13}C in step 6 above, `dn='P31'`, `dm='nnn'`, and `dof` from the following table:

^{1}H frequency (MHz)	*^{13}C (Hz)*	*^{15}N (Hz)*	*^{31}P (Hz)*
400	–965	–12000	9000
300	–9000	–9200	7000

12. To determine the ^{31}P X-nucleus 90 pulse width and rf homogeneity using the HMQC pulse sequence, `pwx` is arrayed for a particular `pwxlvl`. The spectrum corresponding to the^{31}P X-nucleus 90 pulse width using the HMQC pulse sequence is the maximum amplitude spectrum.

    An array of spectra appears as shown in Figure 6. In HMQC, `null` occurs at 45, 135 maximum at 90 and negative at 180 .

    Set `pwxlvl` to the `tpwr` value for normal ^{31}P observe (to get this value you can enter `getparam('tpwr','P31'):pwxlvl`). Enter `array` to array `pwx`. As `array` displays the following prompts, enter in the response shown in bold:

```
parameter to be arrayed:          pwx
number of steps in the array:     20
starting increment:               2
array increment:                  1
```

    In this example, the experiment is set up to array `pwx` from 2 µs to 22 µs, in 1 µs increments.

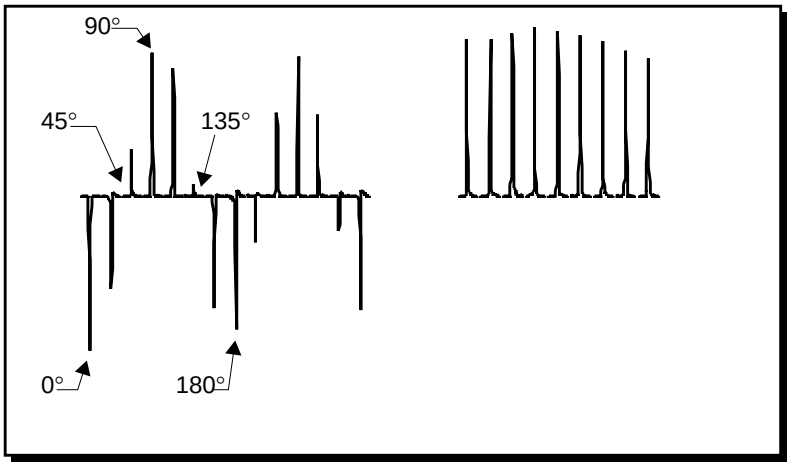

Figure 6. `pwx` calibration, coarse (left) and fine (right)

13. Enter `ga` to start the acquisition. After the last spectrum is finished, enter `ds(1)` to display the first spectrum. Enter `aph` to phase the spectrum, then enter `ai dssh` to display the arrayed spectra.
14. Determine the `pwx90` from the first maximum. Write the results for each probe in the forms provided in "Liquids Probes Test Results" on page 83.
15. To store the values in the probe calibrations file, enter the following commands:

    ```
    setparams('pwxlvl','61.0','P31')
    setparams('pwx','14.2','P31')
    ```

    Be sure to enclose the values in single quotes because they are treated as strings.

^{31}P Decoupling Calibration—Measuring γH_2 for Indirect Detection

1. Set `pwx` equal to the value determined in step 14 in the previous procedure.
2. Enter `ga` to acquire a spectrum.
3. Set `dmm='ccc' dm='nny' dpwr=30 dof=dof+2000,dof-2000`. Then enter `ga`.
4. Enter `ds(1)`, place a cursor on each of the positive peaks, and write down the delta.
5. Enter `ds(2)`, place a cursor on each of the positive peaks, and write down the delta.
6. Enter `h2cal` and enter the delta values for the high field and low field coupling.
7. Enter `10.5` for the coupling constant when prompted.

   A γH_2 of 3000 is necessary for decoupling in indirect detection. If the value is not 3000, increase `dpwr` in increments of 3 dB until γH_2 is 3000.
8. To store the values in the probe calibrations file, enter the following commands:

   ```
   setparams('dpwr','44','P31')
   setparams('dmf','12000','P31')
   ```

   Be sure to enclose the values in single quotes because they are treated as strings.

^{15}N pwx90 Pulse Width (Only 400-MHz with Indirect Detection Probes)

This test is performed at installation only on 400-MHz systems with indirect detection probes, unless explicitly agreed upon in writing as part of the customer contract.

Sample

Sample Tube (mm)	*Test Sample*	*Sample Part Number*
5	2% benzamide–^{15}N, 0.2% Cr(acac) in $CDCl_3$	00-968120-97

Procedure

To perform the ^{15}N or X-nucleus 90 pulse width calibration in the indirect mode, use the HMQC pulse sequence.

1. Make sure the appropriate quarter-wavelength cable and probe tuning rod are installed.
2. Insert the sample. Tune the probe on this sample.
3. Enter `rtp('/vnmr/tests/H1sn')` to retrieve the parameters from the ^{1}H sensitivity measurement.
4. Set `pw` and `tpwr` to the values determined for the ^{1}H sensitivity procedure; you can get these values from the probe calibration file by entering:

   ```
   getparam('tpwr'):tpwr
   getparam('pw90'):pw
   ```

5. Set `gain=10 dn='N15' d1=5` and enter `ga`.
6. Place two cursors around the region with the peaks. Enter `movesw` to narrow the spectral width.
7. Determine the 90 pulse width by arraying `pw` to ≥360 pulse width. The the macro `array` to set up the array. As the macro displays the following prompts, type in the response shown in bold:

   ```
   parameter to be arrayed:          pw
   number of steps in the array:     25
   starting increment:               2
   array increment:                  2
   ```

   In the above example, the experiment is set up to array the `pw` from 2 µs to 52 µs, in 2 µs increments. If the 90 pulse width is 10µs, the 450 pulse width corresponds to `pw=50` µs (5 * `pw90`).
8. Enter `ga` to start the acquisition. After the last spectrum is finished, type `ds(5) vp=50` to display the fifth spectrum. Type `aph` to phase the spectrum, then type `ai dssh` to display the arrayed spectra.
9. Set `pw` equal to the 90 pulse width you have determined. Ether `ga` to acquire the spectrum. Enter `f ds` to display the spectrum.
10. To perform the ^{15}N X-nucleus 90 pulse width calibration in the indirect mode using HMQC, enter `hmqc` in the same experiment or move the parameters to a

different experiment by entering `mp(x,y)`, where `x` is the current experiment and `y` is the experiment to which the parameter set is to be moved.

11. Set the following parameters: `fn=8192 ni=1 phase=1 nt=1 ss=0 spin=0` (use `acqi` to turn the spinner off if the optional spinner hardware is not installed) `d1=2`, and set `pw` to the ^{1}H 90 pulse width(determined from the previous steps), `tpwr` to the `tpwr` level to give the ^{1}H 90 pulse width (determined from the previous steps), `null=0`, `j=90`, `tof` to the `tof` determined from the ^{1}H spectrum of the 2% benzamide-^{15}N in step 6 above, `tn='N15'`, `dm='nnn'`, and `dof` from the following table:

^{1}H frequency (MHz)	*^{13}C (Hz)*	*^{15}N (Hz)*	*^{31}P (Hz)*
400	–965	–12000	9000
300	–9000	–9200	7000

12. To determine the ^{15}N X-nucleus 90 pulse width and rf homogeneity using the HMQC pulse sequence, `pwx` is arrayed for a particular `pwxlvl`. The spectrum corresponding to the ^{15}N X-nucleus 90 pulse width using the HMQC pulse sequence is the maximum amplitude spectrum.

    An array of spectra appears as shown in Figure 7. In HMQC, `null` occurs at 45, 135 maximum at 90 and negative at 180 .

    Set `pwxlvl` to the `tpwr` value for normal ^{15}N observe (to get this value you can enter `getparam('tpwr','N15'):pwxlvl`), Enter `array` to array `pwx`. As `array` displays the following prompts, enter in the response shown in bold:

```
parameter to be arrayed:          pwx
number of steps in the array:     20
starting increment:               2
array increment:                  1
```

    In this example, the experiment is set up to array `pwx` from 2 µs to 22 µs, in 1 µs increments.

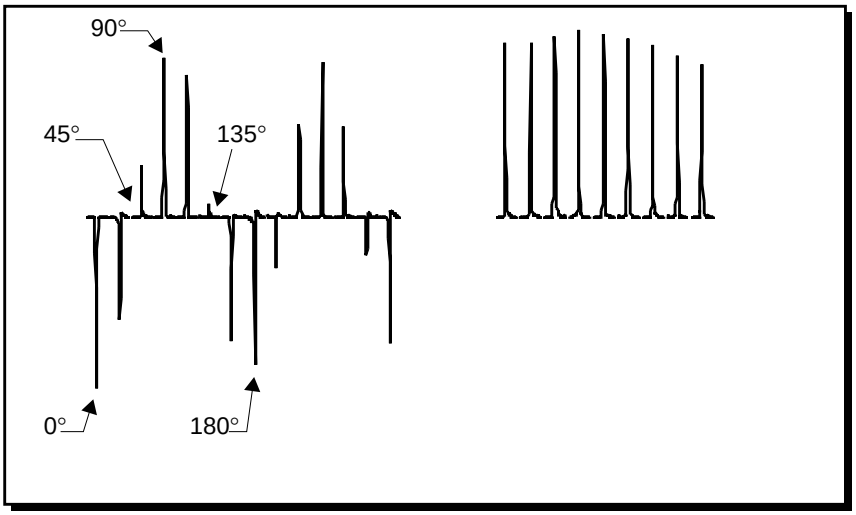

Figure 7. pwx calibration, coarse (left) and fine (right)

13. Enter `ga` to start the acquisition. After the last spectrum is finished, enter `ds(1)` to display the first spectrum. Enter `aph` to phase the spectrum, then enter `ai dssh` to display the arrayed spectra.
14. Determine the `pwx90` from the first maximum. Write the results for each probe in the forms provided in "Liquids Probes Test Results" on page 83.
15. To store the values in the probe calibrations file, enter the following commands:

    ```
    setparams('pwxlvl','61.0','N15')
    setparams('pwx','14.2','N15')
    ```

    Be sure to enclose the values in single quotes because they are treated as strings.

^{15}N Decoupling Calibration—Measuring γH_2 for Indirect Detection

This procedure describes how to perform ^{15}N decoupling calibration as well as how to measure γH_2 for indirect detection.

1. Set `pwx` equal to the value determined in step 14 in the previous procedure.
2. Enter `ga` to acquire a spectrum.
3. Set `dmm='ccc' dm='nny' dpwr=30 dof=dof+2000,dof-2000`. Then enter `ga`.
4. Enter `ds(1)`, place a cursor on each of the positive peaks, and write down the delta.
5. Enter `ds(2)`, place a cursor on each of the positive peaks, and write down the delta.
6. Enter `h2cal` and enter the delta values for the high field and low field coupling.
7. Enter `90` for the coupling constant when prompted.

   A γH_2 of 3000 is necessary for decoupling in indirect detection. If the value is not 3000, increase `dpwr` in increments of 3 dB until γH_2 is 3000.
8. To store the values in the probe calibrations file, enter the following commands:

   ```
   setparams('dpwr','42','N15')
   setparams('dmf','12000','N15')
   ```

   Be sure to enclose the values in single quotes because they are treated as strings.

2.5 Sensitivity Procedures

This section covers the sensitivity procedures required to demonstrate the specifications listed in “Sensitivity Specifications” on page 67.

This section contains the following test procedures:

- 1H Sensitivity
- 19F Sensitivity
- 31P Sensitivity
- 13C Sensitivity
- 29Si Sensitivity (Only 4-Nucleus Probes with 29Si)
- 15N Sensitivity (Only 400-MHz or 4-Nucleus Probes with 15N)

^{1}H Sensitivity

Samples

Sample Tube (mm)	*Test Sample*	*Sample Part No.*
5	0.1% ethylbenzene, 0.01% TMS 99.89% deuterochloroform ($CDCl_3$)	00-968120-70
10	0.1% ethylbenzene, 0.01% TMS 99.89% deuterochloroform ($CDCl_3$)	00-968123-70
4	0.1% ethylbenzene, 0.01% TMS 99.89% deuterochloroform ($CDCl_3$)	00-993143-99

Procedure

1. If you just finished the 90 pulse width calibration, enter `pw=pw90 f full nm` and skip to step 5.
2. Enter `rtp('/vnmr/tests/H1sn') su`.
3. If you completed the 90 pulse width calibration using*GLIDE*, enter the following:

   ```
   getparam('tpwr'):tpwr
   getparam('pw90'):pw
   ```
4. Tune the probe.
5. Enter `nt=1 ga` to acquire the spectrum.
6. When the spectrum is displayed, phase it and enter `wp=500` to display the quartet.
7. Use the cursors to locate a 200-Hz noise region to the left (downfield) of the quartet, as shown in Figure 8.
8. Enter `dsn`. The computer calculates the signal-to-noise ratio.
9. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.
10. After the specifications are met, do the following:
    - Write down the values for `z0`, `lockpower`, and `lockgain` as they appear in the `Acqi` window. Use the forms provided in “Liquids Probes Test Results” on page 83.
    - Save the shims by entering the following command in the VNMR input window:

      ```
      svs('cdcl3')
      ```

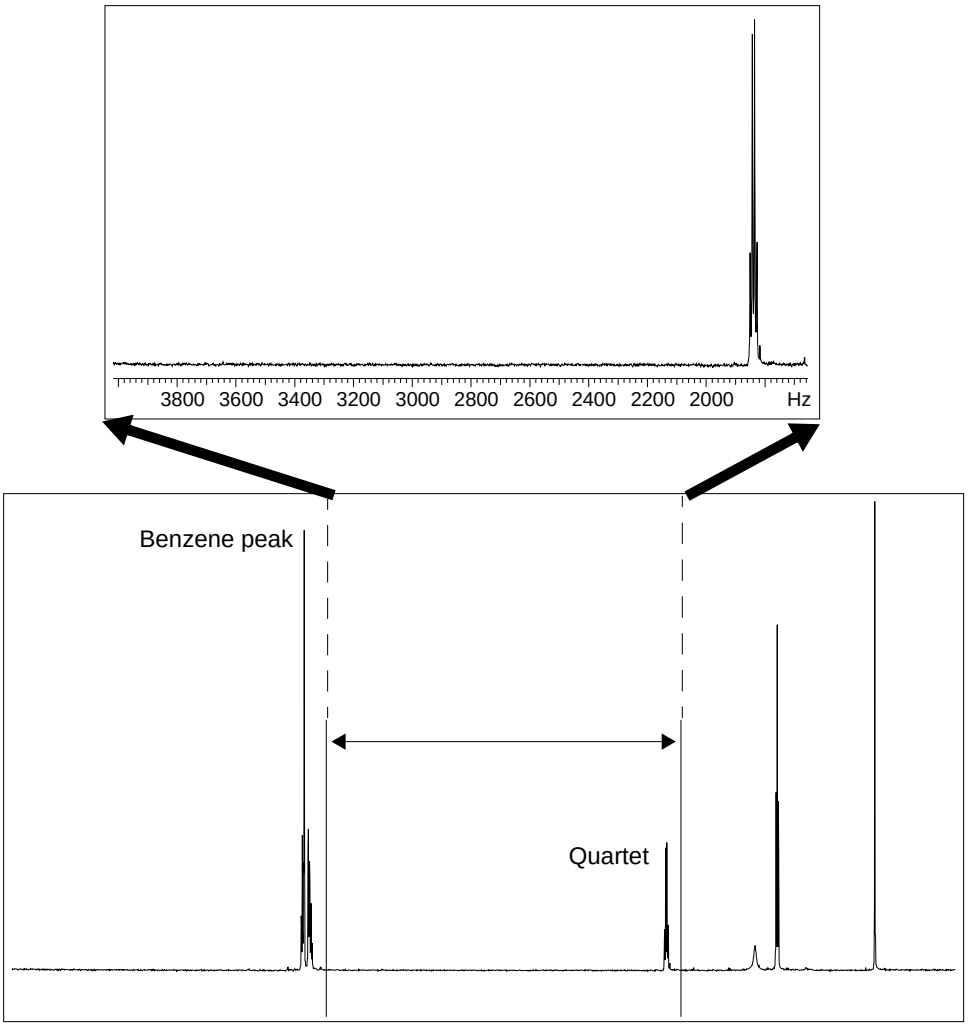

Figure 8. ^{1}H sensitivity measurement

^{19}F Sensitivity

Sample

Sample Tube (mm)	*Test Sample*	*Sample Part No.*
5	0.05% trifluorotoluene in benzene–d_6	00-968120-82

Procedure

1. If you just finished the 90 pulse width calibration, enter `pw=pw90 f full nm` and skip to step 5.
2. Enter `rtp('/vnmr/tests/F19sn') su`.
3. If you completed the 90 pulse width calibration using*GLIDE*, enter the following:

   ```
   getparam('tpwr'):tpwr
   getparam('pw90'):pw
   ```

4. Tune the probe.
5. Enter `lb=1.6 ga` to acquire the spectrum. The spectrum is a single line.
6. Choose a 200-Hz noise region with the cursors, then enter the command `dsn` to determine the signal-to-noise ratio.
7. Retune the probe to ^{1}H for the next test.
8. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

^{31}P Sensitivity

Samples

Sample Tube (mm)	*Test Sample*	*Sample Part No.*
5	0.0485 M triphenylphosphate in $CDCl_3$	00-968120-87
10	0.0485 M triphenylphosphate in $CDCl_3$	00-968123-87

Procedure

1. Make sure the appropriate quarter-wavelength cable is installed.
2. If you just finished the 90 pulse width calibration, enter `pw=pw90 f full nm` and skip to step 5.
3. Enter `rtp('/vnmr/tests/P31sn') su`.
4. If you completed the 90 pulse width calibration using*GLIDE*, enter the following:

   ```
   getparam('tpwr'):tpwr
   getparam('pw90'):pw
   ```

5. Tune the probe.
6. Enter `ga` to acquire the spectrum. The spectrum is a single line.
7. Set `vp=50` and adjust `vs` so that the peak covers about half the screen.
8. For noise measurement, locate the cursor in a representative 2000 Hz region.
9. Enter `dsn`. The computer measures the signal-to-noise ratio.
10. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

^{13}C Sensitivity

Samples

Sample Tube (mm)	*Test Sample*	*Sample Part No.*
5	40% p-dioxane in benzene–d_6 (ASTM)	00-968120-69
10	40% p-dioxane in benzene–d_6 (ASTM)	00-968123-69

Procedure

The specification for the ^{13}C lineshape test must be met before performing this test. For this test you must recall parameters because the ^{13}C pw90 test leaves `pp` calibration parameters.

1. Make sure the appropriate quarter-wavelength cable is installed.
2. Enter `rtp('/vnmr/tests/C13sn') su`.
3. If you completed the 90 pulse width calibration using*GLIDE*, enter the following:

   ```
   getparam('tpwr'):tpwr
   getparam('pw90'):pw
   ```

4. Tune the probe.
5. Enter `nt=1 ga` to acquire the spectrum.

   To ensure that C_6D_6 lines are fully relaxed, wait at least 8 minutes between acquisitions.)
6. The signal of interest is the highest line of the C_6D_6 triplet (left side of display as shown in Figure 9). Use the cursors to select a 1400 Hz wide noise region between the C_6D_6 and dioxane peaks.
7. Enter `dsn`. The computer calculates the signal-to-noise ratio.
8. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.
9. After the specifications are met, do the following:
   - Write down the values for `z0`, `lockpower`, and `lockgain` as they appear in the `Acqi` window. Use the forms provided in “Liquids Probes Test Results” on page 83.
   - Save the shims by entering the following command in the VNMR input window:

     `svs('c6d6')`

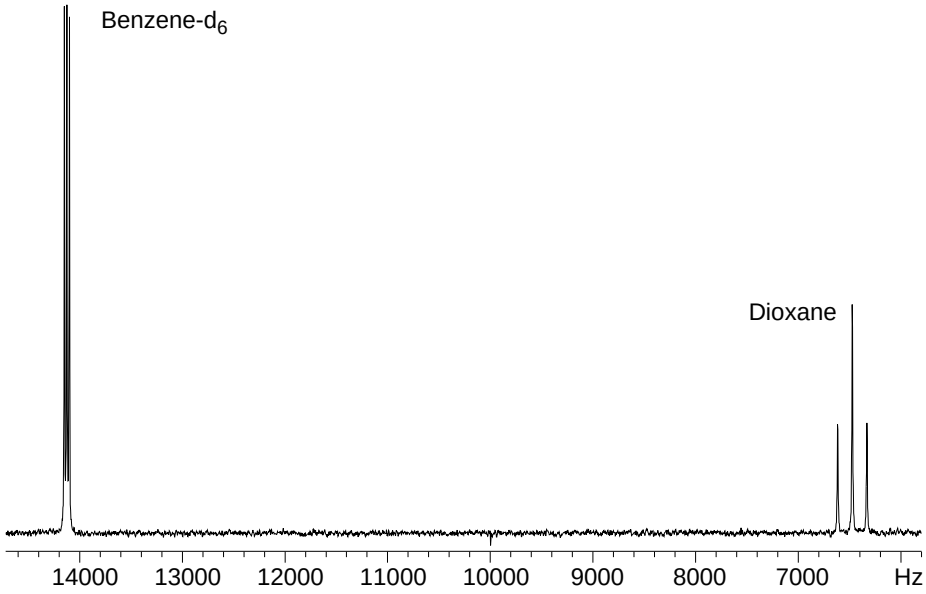

Figure 9. ^{13}C Sensitivity

^{29}Si Sensitivity (Only 4-Nucleus Probes with ^{29}Si)

This test is for broadband systems only.

Sample

Sample Tube (mm)	*Test Sample*	*Sample Part No.*
5	25% hexamethyldisiloxane in benzene–d_6	00-968120-98

Procedure

1. Make sure the appropriate quarter-wavelength cable is installed.
2. Enter `rtp('/vnmr/tests/Si29sn') su`.
3. Tune the probe.
4. Set `dmf` and `dpwr` from the ^{13}C calibration by entering:

   ```
   getparam('dmf','H1'):dmf
   getparam('dpwr','H1'):dpwr
   ```

5. Set `tpwr` and `pw` from the ^{29}Si calibration by entering:

   ```
   getparam('tpwr',):tpwr
   getparam('pw90'):pw
   ```

6. Enter `su`.
7. Enter `fn=16384 dm='nny' ga` to acquire a spectrum. The spectrum is a single line.
8. For noise measurement, locate the cursor in a representative 500 Hz region.
9. Enter `dsn`. The computer calculates the signal-to-noise ratio.
10. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.

^{15}N Sensitivity (Only 400-MHz or 4-Nucleus Probes with ^{15}N)

This test is performed at installation only on 400-MHz systems and broadband systems with 5-mm ^{1}H/^{19}F/^{13}C/^{15}N 4-nucleus probes, unless explicitly agreed upon in writing as part of the customer contract.

Samples

Sample Tube (mm)	*Test Sample*	*Sample Part No.*
5	90% formamide in DMSO–d_6	00-968120-83
10	90% formamide in DMSO–d_6	00-968123-83

Procedure

1. Make sure the appropriate quarter-wavelength cable and probe tuning rod are installed.
2. If you just finished the 90 pulse width calibration, enter `pw=pw90 f full` and skip to step 5.
3. Enter `rtp('/vnmr/tests/N15sn') su`.
4. If you completed the 90 pulse width calibration and entered the values into the probe calibration file, you can retrieve these values by entering:

   ```
   getparam('tpwr'):tpwr
   getparam('pw90'):pw
   ```

5. Tune the probe.
6. For a 5-mm sample, set `nt=4`. For a 10-mm sample, set `nt=1`.
7. Set `dmf` and `dpwr` from the calibration by entering:

   ```
   getparam('dmf','H1'):dmf
   getparam('dpwr','H1'):dpwr
   ```

8. Enter `lb=0.4 fn=16384 d1=100 dm='nny' ga` to acquire s spectrum. the spectrum is a single line.
9. For noise measurement, locate the cursor in a representative 100 Hz region.
10. Enter `dsn`. The computer calculates the signal-to-noise ratio.
11. Write the results for each probe in the forms provided in “Liquids Probes Test Results” on page 83.
12. After the specifications are met, do the following:
    - Write down the values for `z0`, `lockpower`, and `lockgain` as they appear in the `Acqi` window. Use the forms provided in “Liquids Probes Test Results” on page 83.
    - Save the shims by entering the following command in the VNMR input window:

      ```
      svs('dmso')
      ```

2.6 Configuring Solvent-Based Shims and setlk for *GLIDE*

For *GLIDE* and automation to work properly, solvent-based shims must be set up and the setlk macro must be configured.

To Set Up the Solvent-Based Shim Files

During the probe acceptance tests (ATP), at least three solvent-based shim files were saved: `acetone`, `cdcl3`, and `c6d6` (dmso would have been saved for a 400-MHz system or with a probe with ^{15}N). Other solvents that *GLIDE* supports by default are D_2O and CD_3OD. The steps below describe how to add other solvent-based shims and how to move the shim files to the `/vnmr/shims` directory. You must still be logged in as `vnmr1`. Note that starting with VNMR 6.1, the names for shim files should be all lower case.

1. If you primarily use only the solvents used in the ATP (Acetone, $CDCl_3$, and C_6D_6), skip to step 2 below. If you normally use D_2O or CD_3OD (or DMSO and it wasn't used in the ATP) in addition to the solvents used in the ATP set up the shim files for these solvents as follows:
   a. Lock and shim on each solvent.
   b. Write down the values for `z0`, `lockpower`, and `lockgain` for each solvent. These values are displayed in the `Acqi` window.
   c. Save the shims with the `svs` command in the VNMR input window. Use the following commands:
      - `svs('d2o')` if the solvent is D_2O
      - `svs('cd3od')` if the solvent is CD_3OD
      - `svs('dmso')` if the solvent is DMSO.
2. In a shell tool or a Terminal window (still logged in as `vnmr1`), move the shim files from `~vnmrsys/shims` to `/vnmr/shims`. Change to the `/vnmr/shims` directory and use the `mv` command to move the shim files. Don't forget the dot at the end of each `mv` command.

```
cd /vnmr/shims
mv ~/vnmrsys/shims/acetone .
mv ~/vnmrsys/shims/cdcl3 .
mv ~/vnmrsys/shims/c6d6 .
```

And if present,

```
mv ~/vnmrsys/shims/dmso .
mv ~/vnmrsys/shims/d2o .
mv ~/vnmrsys/CD3OD .
```

Now go to the next section to configure the `setlk` macro.

To Configure the setlk Macro

A properly configured `setlk` is critical to Autolock and Autoshim performance and *GLIDE* performance. The `setlk` macro, once properly configured, allows the system to perform the following adjustments for each system:

- Explicitly set `z0`, `lockpower`, and `lockgain`
- Retrieve solvent-based shim sets

Explicitly setting `z0`, `lockpower`, and `lockgain` for solvents typically used ensures the best possible results when using Autolock. The reliability of Autolock is especially important for the first sample of an automation run—if lock power has been low, the first sample may not be run because of an Autolock failure.

Use the following steps to configure the `setlk` macro.

1. Using a text editor, such as `vi`, open the file `/vnmr/maclib/setlk`.
2. To allow the system to automatically retrieve a set of shims for each solvent, remove the quotes around the following lines:

```
if ($e>0.5)or($e1>0.5 then
  rts($solv)
else
  exists(userdir+'/shims/cdcl3','file'):$e
  exists(systemdir+'/shims/cdcl3','file'):$e1
  if (($e>0.5)or($e1>0.5) then
    rts('cdcl3')
  endif
endif
```

3. Remove the quotes from the lines with the solvents you plan to use. Replace the appropriate values of `z0`, `lockpower`, `lockgain` using the values you wrote down during the ATP or when you locked and shimmed on other solvents.

   The example below shows some of the entries with the Z0 value entered. The Z0 value represents the value of Z0 shown in the lock display in `acqi`.

```
if $solv='cdcl3' then z0=512 lockpower=37 lockgain=41
else
if $solv='d2o' then z0=521 lockpower=37 lockgain=48 else
if $solv='acetone' then z0=512 lockpower=21 lockgain=39
else
if $solv='dmso' then lockpower=33 lockgain=36 else
if $solv='c6d6' then lockpower=20 lockgain=39 else
if $solv='cd3od' then lockpower=25 lockgain=35 else
lockpower=30 lockgain=40
endif endif endif endif endif endif
```

   Note that you should have one `endif` for each `if` in the entries.

4. Save the file and exit the text editor. Clean up the VNMR file system as necessary.

Chapter 3. Console and Magnet Test Procedures

This chapter contains the procedures required to demonstrate the specifications for ***MERCURY*** consoles and magnets. Below is an outline of the tests in this section.

- GLIDE Operation Demonstration
- APT and DEPT Demonstration
- Homonuclear Decoupling (Optional)
- Variable Temperature Operation (Optional)
- Magnet Drift Test

Write the results of the tests in the appropriate forms provided in "Console and Magnet Test Results" on page 87.

GLIDE Operation Demonstration

Samples

Sample Tube (mm)	*Nucleus*	*Test Sample*	*Sample Part Number*
5	^{1}H	0.1% ethylbenzene in $CDCl_3$	00-968120-70
5	^{1}H	0.1% 3-heptanone in $CDCl_3$	00-968120-93
5	^{13}C	40% p-dioxane in benzene–d_6 (ASTM)	00-968120-69
5	^{13}C	10% menthol in $CDCl_3$	00-968120-94

Procedure

1. With the 3-heptanone or the ethylbenzene sample, run fully automated *GLIDE*, which should take about 5 minutes:
   a. Insert the sample.
   b. Click the Setup icon.
   c. In the dialog box that appears, select Proton 1D from the Experiment menu, and then select CDCl3 from the Solvent menu.
   d. Insert the sample. If the Insert/Eject selections are available in the Setup window, click on Insert. Otherwise, use the button on the magnet leg.
   e. Enter the relevant text in the Text field (e.g., standard proton 1D).
   f. Click the Setup button
   g. Click the Go icon in the *GLIDE* window.

      You should get one spectrum displayed and plotted.
2. With the menthol sample, run a carbon spectrum, using Customize to reset the number of scans to 16.
   a. Click the Setup icon.
   b. In the dialog box that appears, select Carbon 1D from the Experiment menu and CDCl3 from the Solvent menu.
   c. Insert the sample. If the Insert/Eject selections are available in the Setup window, click on Insert. Otherwise, use the button on the magnet leg.
   d. Enter the relevant text in the Text field (e.g., custom proton 1D).
   e. Click the Setup button.
   f. Click the Customize icon in *GLIDE*.
   g. Click the Acquire icon in the Custom window.
   h. Scroll through the list of adjustables and enter **16** for the Number of scans.
   i. Click the Close button in the Acquire dialog box.
   j. Click on the up arrow under the Customize icon.
   k. Click the Go icon in the *GLIDE* window.

      You should get one spectrum displayed and plotted.

APT and DEPT Demonstration

The APT (Attached Proton Test) and DEPT (Distortionless Enhancement by Polarization Transfer) spectral editing experiments give an example of the capabilities of the ***MERCURY*** system.

Sample

Sample Size (mm)	*Test Sample*	*Sample Part Number*
5	30% menthol in $CDCL_3$	00-968120-94

Procedure

1. Open *GLIDE* by clicking on the GLIDE button in the VNMR menu.
2. Click the Setup icon.
3. Insert the sample. If the Insert/Eject selections are available in the Setup window, click on Insert. Otherwise, use the button on the magnet leg.
4. Select APT or DEPT as follows:
   - For APT, select APT in the Experiment drop-down menu.
   - For DEPT, select C13 and dept spectra in the Experiment drop-down menu.
5. Select CDCL3 in the Solvent drop-down menu.
6. Enter the relevant text in the Text field (e.g., APT or DEPT).
7. Click the Setup button at the bottom of the Setup window.
8. Click the Go icon and wait until the spectra is plotted.

   The APT spectrum should appear with three peaks upright (CH_2) and seven peaks inverted (CH, CH_3).

   The DEPT experiment performs an automated DEPT analysis (ADEPT), which produces four spectra.

Homonuclear Decoupling (Optional)

This test is only performed on systems with the homonuclear decoupling accessory installed. We recommend using a 5-mm probe capable of ^{1}H direct observe.

Samples

Sample Tube (mm)	*Test Sample*	*Sample Part Number*
5	0.1% ethylbenzene, 0.01% TMS, 99.89% deuterochloroform ($CDCl_3$)	00-968120-70
10	0.1% ethylbenzene, 0.01% TMS, 99.89% deuterochloroform ($CDCl_3$)	00-968123-70

Procedure

1. Enter `rtp('/vnmr/tests/H1sn')`.
2. Tune the probe. Set `nt=1`. Run a normal spectrum without decoupling.
3. Set `dm='yyy'`. Use the cursor and `sd` to set the decoupler on the central line of the triplet, and then run a decoupled spectrum.

   The best values of `dpwr` must be found by experiment. Too much power might show increased noise; too little might not decouple the quartet.

   Setting `dpwr=25` is a good starting point.

   Possible `dpwr` values are 0 to 49 (49 is maximum power), in steps of 1.0 dB.
4. Observe that the quartet collapses to a single peak with no remaining evidence of splitting.

Variable Temperature Operation (Optional)

This optional test demonstrates that the basic variable temperature (VT) unit and probe changes to the desired temperature as registered on the window of the VT controller. If the system is equipped with a VT unit, you should read through the VT operation instructions before this demonstration.

Dry nitrogen is required as the VT gas if the requested temperature is over 100° C or below 10° C. Otherwise, air can be used. Dry nitrogen gas is recommended for cooling the bearing, spinner, and decoupler. This prevents moisture condensation in the probe and spinner housing.

CAUTION: **The use of air as the VT gas for temperatures above 100° C is not recommended. Such use destructively oxidizes the heater element and the thermocouple.**

Demonstration Limitations

If dry nitrogen gas and liquid nitrogen are not available at the time of installation, the range of VT demonstration is limited to temperatures between 30°C and 100°C.

Sample

No sample is used.

Probe and Hardware Requirements

Any variable temperature probe is used.

Basic Specifications

The specifications for variable temperature ranges are listed with each probe in the liquids probes chapter of the *NMR Probes Installation Manual*.

Procedure

1. In the CONFIG window, make sure VT Controller is set to Present. Alternatively, enter `vttype?` to check that `vttype=2`.
2. Set N_2 gas flow to 9.5 to 10.0 LPM (for temperatures below –100° C, increase N_2 flow to 12 LPM).
3. Enter a value for `temp`, then enter `su`. For values below room temperature, the heat exchanger must be in place. Maintain the temperature for 5 minutes.
4. Operate the VT unit within the specifications of the probe. Test the temperature at set points that correspond to the following:
   - Maximum, minimum, and midpoint of the allowed temperature—95, 80, 60 if air is used; 120, 30, 20 if dry nitrogen is used; 120, –100, 40 if a heat exchanger is used.
   - Ambient temperature.

   Be sure to never ramp the temperature by more than 12°C per minute up or down. For probes with small temperature ranges, ramping increments will be much less than 12°C per minute. To achieve the 12°C per minute change, adjust the temperature by no more than 50°C, enter `su`, and wait for the temperature to equilibrate.

Magnet Drift Test

The magnet drift test is an overnight test.

Sample

Use ^{1}H lineshape sample 20% $CHCl_3$ in 80% deuteroacetone $(CD_3)_2CO$, Part No. 00-968120-76.

Probe and Hardware Requirements

We recommend using a 5-mm probe capable of ^{1}H direct observe.

Test Procedure

1. Enter `rtp('/vnmr/tests/H1lshp')` to retrieve the test parameter set to the current experiment.
2. Tune the probe.
3. Acquire a normal spectrum and shim the chloroform signal to less than 1 Hz linewidth at 50%.
4. Connect to the `acqi` window, turn the lock off, turn the spinner off, and set the spinner speed to 0. Make sure the lock signal is on resonance (the lock signal display should be flat). Disconnect the `acqi` window. Then disconnect the lock cable from the probe.
5. Enter `in='n' spin='n' nt=1 array('d1',11,3600,0) d1[1]=60`.
6. This will set up an array of `d1` values, with the first spectrum to be collected after 1 minute and subsequent spectra to be collected at one-hour intervals.
7. Enter `ga` to acquire the spectra.
8. The test takes approximately 10 to 11 hours to finish.
9. Phase the first spectrum by entering `ds(1)` to display the first spectrum of the array and by entering `aph0` to apply a first-order phase correction to the spectrum.
10. Enter `ai` to scale all of the spectra to the same vertical scale, and enter `dssa` to display the arrayed spectra stacked vertically.
11. Compare the frequency shift of the chloroform peak of the arrayed spectra to the frequency of the first spectrum in the array.

Part 2:

Acceptance Test Specifications

Chapter 4 **Liquids Probes Specifications**

Chapter 4. Liquids Probes Specifications

This chapter contains the following specifications for liquids probes:

- 4.1 “Resolution, Lineshape, Spinning Sidebands Specifications,” page 62.
- 4.2 “90 Pulse Width and gH2 Specifications,” page 65.
- 4.3 “Sensitivity Specifications,” page 67.
- 4.4 “Variable Temperature Range Specifications,” page 69.
- 4.5 “Magnet Drift,” page 71.

4.1 Resolution, Lineshape, Spinning Sidebands Specifications

Resolution and lineshape specifications are listed in Table 5 for broadband systems or in Table 6 for 4-nucleus systems. Probes are categorized by frequency, and specifications are listed under the appropriate nucleus.

Table 7 (broadband systems) and Table 8 (4-nucleus systems) list the spinning sidebands acceptance test specifications for Varian liquids probes. Probes are categorized by frequency, and specifications are listed under the appropriate nucleus.

About Resolution and Lineshape Specifications

All resolution, lineshape, and spinning sidebands values of Varian probes should be less than or equal to the values listed in the table. Acceptance test specifications are achieved using the procedures in "Resolution, Lineshape, and Spinning Sidebands Procedures" on page 20.

Blank spaces (shown as "—") in the specifications column indicate that Varian does not specify a value or that it is not applicable to specify a value. Linewidths for resolution and lineshape are in units of Hz, at the 50%, 0.55%, and 0.11% amplitude levels.

The ^{1}H natural lineshape and linewidth of chloroform at high field have a contribution from the spread in chemical shift of ^{35}Cl and ^{37}Cl isotopomers—see Anet and Kopelevich, *J. Am. Chem. Soc.*, **109**, 5870 (1987).

Lineshape for ^{13}C at the 0.55% and 0.11% amplitude levels with 5-mm probes is typically 3/6 spinning and is not demonstrated at installation.

Samples for Resolution and Lineshape Tests

Table 3 lists the samples used to achieve the ^{1}H resolution and lineshape specifications. Table 4 lists the samples used to achieve ^{13}C resolution and lineshape specifications. The 4-mm sample tube has a 4-mm outside diameter (O.D.) and a 40-µL sample volume.

Table 3. Samples for ^{1}H resolution, lineshape, and spinning sidebands tests

Amplitude Level	*Sample Tube (mm)*	*Test Sample*	*Sample Part No.*
50.0%, 0.55%, 0.11%	5	20% chloroform in acetone–d_6	00-968120-76
50.0%, 0.55%, 0.11%	5	5% chloroform in acetone–d_6	00-968120-99
50.0%, 0.55%, 0.11%	4	5% chloroform in acetone–d_6	00-993143-99
50.0%, 0.55%, 0.11%	5	1% chloroform in acetone–d_6	00-968120-89

Table 4. Samples for ^{13}C resolution, lineshape, and spinning sidebands tests

Amplitude Level	*Sample Tube (mm)*	*Test Sample*	*Sample Part No.*
50.0%, 0.55%, 0.11%	5	40% p-dioxane in benzene–d_6 (ASTM)	00-968120-69
50.0%, 0.55%, 0.11%	10	40% p-dioxane in benzene–d_6 (ASTM)	00-968123-69

Table 5. Resolution and lineshape specifications for broadband systems

Probe	*Resolution and Linewidth* * *(Hz)*	
200-MHz	*^{1}H Spinning*	*^{13}C Spinning*
5-mm ^{15}N-^{31}P Broadband	—	0.2
10-mm ^{15}N-^{31}P Broadband	—	0.2
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable	0.4/6.0/12.0	0.2
300-MHz	*^{1}H Spinning*	*^{13}C Spinning*
5-mm -^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	0.4/6.0/12.0	0.2
10-mm ^{15}N-^{31}P Broadband	—	0.2
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection	0.4/6.0/12.0	0.2
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection PFG	0.4/6.0/12.0	0.2
4-mm (40 µL) ^{1}H Nano	Available by request.	
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable/Broadband	0.4/6.0/12.0	0.2
400-MHz	*^{1}H Spinning*	*^{13}C Spinning*
5-mm -^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	Available by request.	
10-mm ^{15}N-^{31}P Broadband	—	0.2
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection	0.45/6.0/12.0	—
5-mm -^{1}H{^{15}N–^{31}P} Indirect Detection PFG	0.45/6.0/12.0	0.2
4-mm (40 µL) ^{1}H Nano	Available by request.	
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable	0.45/6.0/12.0	0.2

**. Full linewidth at half height.*

Table 6. Resolution and lineshape specifications for 4-nucleus systems

Probe	*Resolution and Linewidth* * *(Hz)*	
200-MHz	*^{1}H Spinning*	*^{13}C Spinning*
5-mm ^{1}H/^{13}C RT Computer-Switchable	0.4/6.0/12.0	0.2
5-mm ^{1}H/^{13}C VT Computer-Switchable	0.4/6.0/12.0	0.2
300-MHz	*^{1}H Spinning*	*^{13}C Spinning*
5-mm -^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	0.4/6.0/12.0	—
5-mm ^{1}H/^{13}C RT Computer-Switchable	0.4/6.0/12.0	0.2
5-mm ^{1}H/^{13}C VT Computer-Switchable	0.4/6.0/12.0	0.2
4-mm (40 µL) ^{1}H Nano	Available by request.	

**. Full linewidth at half height.*

Table 7. Spinning sidebands specifications for broadband systems

Probe	*Spinning Sidebands*	
200 MHz	*^{1}H*	*^{13}C*
5-mm ^{15}N-^{31}P Broadband	—	1%
10-mm ^{15}N-^{31}P Broadband	—	1%
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable	1%	1%
300 MHz	*^{1}H*	*^{13}C*
5-mm ^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	1%	1%
10-mm ^{15}N-^{31}P Broadband	—	1%
5-mm $^{1}H\{^{15}N$-$^{31}P\}$ Indirect Detection	1%	1%
5-mm $^{1}H\{^{15}N$-$^{31}P\}$ Indirect Detection PFG	1%	1%
4-mm (40 µL) ^{1}H Nano	Available by request.	
5-mm ^{1}H/^{19}F Proton	1%	—
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable/Broadband	1%	1%
400 MHz	*^{1}H*	*^{13}C*
5-mm ^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	Available by request.	
10-mm ^{15}N-^{31}P Broadband	—	1%
5-mm $^{1}H\{^{15}N$-$^{31}P\}$ Indirect Detection	1%	—
5-mm $^{1}H\{^{15}N$–$^{31}P\}$ Indirect Detection PFG	1%	—
4-mm (40 µL) ^{1}H Nano	Available by request.	
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable	1%	1%

Table 8. Spinning sidebands specifications for 4-nucleus systems

Probe	*Spinning Sidebands*	
200 MHz	*^{1}H*	*^{13}C*
10-mm ^{15}N-^{31}P Broadband	—	1%
5-mm ^{1}H/^{13}C RT Computer-Switchable	1%	1%
5-mm ^{1}H/^{13}C VT Computer-Switchable	1%	1%
300 MHz	*^{1}H*	*^{13}C*
5-mm ^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	1%	1%
5-mm ^{1}H/^{13}C RT Computer-Switchable	1%	1%
5-mm ^{1}H/^{13}C VT Computer-Switchable	1%	1%
4-mm (40 µL) ^{1}H Nano™	Available by request.	

4.2 90 Pulse Width and γH_2 Specifications

The 90 pulse width and γH_2 acceptance test specifications for Varian liquids probes are listed in Table 10 for broadband systems or in Table 11 for 4-nucleus systems. Probes are categorized by frequency, and specifications are listed under the appropriate nucleus.

About 90 Pulse Width and γH_2 Specifications

All 90° pulse width and γH_2 values of Varian probes should be less than or equal to the values listed in the table. Acceptance test specifications are achieved using the procedures in the section "90 Pulse Width Procedures" on page 25.

Blank spaces (shown as "—") in the specifications column indicate that Varian does not specify a value or that it is not applicable to specify a value. Asterisks (*) in the specifications column indicate that the 90 pulse width was determined using the indirect method. Refer to the probe installation manuals for the power-handling capability of Varian probes.

About Test Samples

Table 9 lists the samples used to achieve the 90 pulse width and γH_2 specifications. Note that the 4-mm sample tube is used only for Nano probes. The 4-mm sample tube has a 4-mm outside diameter (O.D.) and a 40-µL sample volume. Doped D_2O can be used for the ^1H pulse width test instead of the sample specified in Table 9.

Table 9. Samples for 90 pulse width and γH_2 tests

Nucleus	*Sample Tube (mm)*	*Test Sample*	*Sample Part Number*
^1H	5	0.1% ethylbenzene, 0.01% TMS 99.89% deuterochloroform ($CDCl_3$)	00-968120-70
^1H	4	0.1% ethylbenzene, 0.01% TMS 99.89% deuterochloroform ($CDCl_3$)	00-993143-99
^{19}F	5	0.05% trifluorotoluene in benzene–d_6	00-968120-82
^{31}P	5	0.0485 M triphenylphosphate in $CDCl_3$	00-968120-87
^{31}P	10	0.0485 M triphenylphosphate in $CDCl_3$	00-968123-87
^{13}C	5	40% p-dioxane in benzene–d_6 (ASTM)	00-968120-69
^{13}C (indirect)	5	1% iodomethane–^{13}C in $CDCl_3$	00-968120-96
^{13}C	10	40% p-dioxane in benzene–d_6 (ASTM)	00-968123-69
^{15}N	5	90% formamide in DMSO–d_6	00-968120-83
^{15}N (indirect)	5	2% benzamide–^{15}N in DMSO–d_6	00-968120-97
^{15}N	10	90% formamide in DMSO–d_6	00-968123-83
^{29}Si	5	25% hexamethyldisiloxane in benzene–d_6	00-968120-98

Table 10. 90° pulse width and γH_2 specifications for broadband systems

Probe	*90° Pulse Width Specifications (µs)*						γH_2
200 MHz	*^{1}H*	*^{19}F*	*^{31}P*	*^{13}C*	*^{15}N*	*^{29}Si*	*(Hz)*
5-mm ^{15}N-^{31}P Broadband	—	—	—	15	—	—	2700
10-mm ^{15}N-^{31}P Broadband	—	—	20	20	—	—	2700
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable	20	20	—	15	—	—	2700
300 MHz	*^{1}H*	*^{19}F*	*^{31}P*	*^{13}C*	*^{15}N*	*^{29}Si*	
5-mm ^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	12	18	15	12	—	—	2700
10-mm ^{15}N-^{31}P Broadband	—	—	20	20	—	—	2700
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection	15	—	—	18	—	—	2700
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection PFG	10	—	—	18	—	—	2700
4-mm (40 µL) ^{1}H Nano	Available by request.						2700
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable/Broadband	12	12	—	15	—	—	2700
400 MHz	*^{1}H*	*^{19}F*	*^{31}P*	*^{13}C*	*^{15}N*	*^{29}Si*	
5-mm ^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	Available by request.						
10-mm ^{15}N-^{31}P Broadband	—	—	—	20	—	—	2700
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection	10	—	—	18*	—	—	2700
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection PFG	10	—	—	18	—	—	2700
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable	20	15	15	15	15	—	2700

**. 90° pulse width determined using the indirect method.*

Table 11. 90° pulse width and γH_2 specifications for 4-nucleus systems

Probe	*90° Pulse Width Specifications (µs)*				γH_2
200 MHz	*^{1}H*	*^{19}F*	*^{31}P*	*^{13}C*	*(Hz)*
5-mm ^{1}H/^{13}C RT Computer-Switchable	18	—	—	18	2700
5-mm ^{1}H/^{13}C VT Computer-Switchable	18	—	—	18	2700
300 MHz	*^{1}H*	*^{19}F*	*^{31}P*	*^{13}C*	
5-mm ^{1}H/^{19}F/^{13}C/^{31}P PFG AutoSwitchable	18	25	15	15	2700
5-mm ^{1}H/^{13}C RT Computer-Switchable	18	—	—	18	2700
5-mm ^{1}H/^{13}C VT Computer-Switchable	18	—	—	18	2700
4-mm (40 µL) ^{1}H Nano	Available by request.				2700

4.3 Sensitivity Specifications

Table 13 (broadband systems) and Table 14 (4-nucleus systems) list the sensitivity acceptance test specifications for Varian liquids probes. Probes are categorized by frequency, and specifications are listed under the appropriate nucleus.

About Sensitivity Specifications

Sensitivity values of varian probes should be greater than or equal to the values listed in the table. Acceptance test specifications are achieved using the procedures in the section "Sensitivity Procedures" on page 42.

Tests are performed in 5-mm sample tubes with 0.38-mm wall thickness (Wilmad 528-PP, or equivalent) and 10-mm sample tubes with 0.46-mm wall thickness (Wilmad 513-7PP, or equivalent). Use of sample tubes with thinner walls (Wilmad 5-mm sample tubes 545-PPT, or equivalent; Wilmad 10-mm sample tubes 513-7PPT, or equivalent) will increase signal-to-noise.

Blank spaces (shown as "—") in the specifications column indicate that Varian does not specify a value or that it is not applicable to specify a value.

If a pulse width is not provided but is needed to achieve a sensitivity acceptance test specification, calibrate using the test results obtained in the factory.

About Test Samples

Table 12 lists the samples used to achieve the sensitivity specifications. Note that the 4-mm sample tube is used only for Nano probes. The 4-mm sample tube has a 4-mm outside diameter (O.D.) and a 40-µL sample volume.

Table 12. Samples for sensitivity tests

Nucleus	*Sample Tube (mm)*	*Test Sample*	*Sample Part Number*
^{1}H	5	0.1% ethylbenzene in $CDCl_3$	00-968120-70
^{1}H	4	0.1% ethylbenzene in $CDCl_3$	00-993143-99
^{19}F	5	0.05% trifluorotoluene in benzene–d_6	00-968120-82
^{31}P	5	0.0485 M triphenylphosphate in $CDCl_3$	00-968120-87
^{31}P	10	0.0485 M triphenylphosphate in $CDCl_3$	00-968123-87
^{13}C	5	40% p-dioxane in benzene–d_6 (ASTM)	00-968120-69
^{13}C	10	40% p-dioxane in benzene–d_6 (ASTM)	00-968123-69
^{15}N	5	90% formamide in DMSO–d_6	00-968120-83
^{15}N	10	90% formamide in DMSO–d_6	00-968123-83
^{29}Si	5	25% hexamethyldisiloxane in benzene–d_6	00-968120-98

Table 13. Sensitivity (S/N) specifications for broadband systems

Probe	*Sensitivity (S/N) Specifications*					
200 MHz	*^{1}H*	*^{19}F*	*^{31}P*	*^{13}C*	*^{15}N*	*^{29}Si*
5-mm ^{15}N-^{31}P Broadband	—	—	40:1	45:1	—	—
10-mm ^{15}N-^{31}P Broadband	—	—	125:1	140:1	—	—
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable	40:1	40:1	40:1	40:1	—	—
300 MHz	*^{1}H*	*^{19}F*	*^{31}P*	*^{13}C*	*^{15}N*	*^{29}Si*
5-mm ^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG						
4-nucleus mode	105:1	105:1	70:1	80:1	—	—
switchable mode	105:1	105:1	100:1	90:1	—	—
10-mm ^{15}N-^{31}P Broadband	—	—	250:1	280:1	—	—
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection	135:1	—	—	—	—	—
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection PFG	180:1	—	—	—	—	—
4-mm (40 µL) ^{1}H Nano	Available by request.					
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable/Broadband	130:1	130:1	105:1	105:1	—	—
400 MHz	*^{1}H*	*^{19}F*	*^{31}P*	*^{13}C*	*^{15}N*	*^{29}Si*
5-mm ^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	Available by request.					
10-mm ^{15}N-^{31}P Broadband	—	—	350:1	450:1	45:1	—
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection	350:1	—	—	—	—	—
5-mm ^{1}H{^{15}N–^{31}P} Indirect Detection PFG	350:1	—	—	—	—	—
4-mm (40 µL) ^{1}H Nano	Available by request.					
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable	130:1	130:1	150:1	120:1	15:1	—

Table 14. Sensitivity (S/N) specifications for 4-nucleus systems

Probe	*Sensitivity (S/N) Specifications*			
200 MHz	*^{1}H*	*^{19}F*	*^{31}P*	*^{13}C*
5-mm ^{1}H/^{13}C RT Computer-Switchable	30:1	—	—	35:1
5-mm ^{1}H/^{13}C VT Computer-Switchable	30:1	—	—	35:1
300 MHz	*^{1}H*	*^{19}F*	*^{31}P*	*^{13}C*
5-mm ^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG				
4-nucleus mode	105:1	105:1	70:1	75:1
switchable mode	105:1	105:1	100:1	90:1
5-mm ^{1}H/^{13}C RT Computer-Switchable	60:1	—	—	70:1
5-mm ^{1}H/^{13}C VT Computer-Switchable	130:1	—	—	105:1
4-mm (40 µL) ^{1}H Nano™	Available by request.			

4.4 Variable Temperature Range Specifications

Table 15 (broadband systems) and Table 16 (4-nucleus systems) list the variable temperature (VT) acceptance tests specifications for Varian liquids probes. Probes are categorized by frequency, and specifications are listed under the appropriate nucleus.

About VT Range Specifications

Acceptance test specifications are achieved using the procedure in "Variable Temperature Operation (Optional)" on page 57. To achieve the VT range specifications, the probe is tested empty.

Table 15. VT range specifications for broadband systems

Probes	*VT Range (°C)*
200 MHz	
5-mm ^{15}N-^{31}P Broadband	–150 to +200
10-mm ^{15}N-^{31}P Broadband	–150 to +200
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable	–150 to +200
300 MHz	
5-mm ^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	–20 to +80
10-mm ^{15}N-^{31}P Broadband	–150 to +200
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection	–100 to +100
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection PFG	0 to +50
4-mm (40 µL) Nano	Room temperature.
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable/Broadband	–100 to +160
400 MHz	
5-mm ^{1}H/^{19}F/^{13}C/^{31}P AutoSwitchable PFG	Available by request.
10-mm ^{15}N-^{31}P Broadband	–150 to +200
5-mm ^{1}H{^{15}N-^{31}P} Indirect Detection	–100 to +100
5-mm 1H{15N–31P} Indirect Detection PFG	0 to +50
4-mm (40 µL) Nano	Room temperature.
5-mm ^{1}H/^{19}F/^{15}N-^{31}P Switchable	–150 to +200

Table 16. VT range specifications for 4-nucleus systems

Probes	*VT Range (°C)*
200 MHz	
5-mm $^{1}H/^{13}C$ RT Computer-Switchable	Room temperature.
5-mm $^{1}H/^{13}C$ VT Computer-Switchable	–150 to +200
300 MHz	
5-mm $^{1}H/^{19}F/^{13}C/^{31}P$ AutoSwitchable PFG	–20 to +80
5-mm $^{1}H/^{13}C$ RT Computer-Switchable	Room temperature.
5-mm $^{1}H/^{13}C$ VT Computer-Switchable	–100 to +160
4-mm (40 µL) ^{1}H Nano	Room temperature.

4.5 Magnet Drift

Table 17 lists the drift specifications for magnets. Specifications for nominal field decay rate are less than or equal to the values listed in the table.

About Magnet Drift Specifications

Use ^{1}H lineshape sample 20% $CHCl_3$ in 80% deuteroacetone $(CD_3)_2CO$, Part No. 00-968120-76. We recommend using a 5-mm probe capable of ^{1}H direct observe.

Table 17. Magnet Drift Specifications

System (MHz/mm)	*Field Strength (T)*	*Nominal Field Decay Rate (Hz/hr)*
200/54, 200/89	4.70	2
300/54, 300/89	7.05	3
400/54	9.40	8
400/89	9.40	10

Part 3:

Acceptance Test Results

Chapter 5 **Acceptance Test Results**

Chapter 5. Acceptance Test Results

This chapter contains the following forms for recording system information and acceptance test results:

- 5.1,“Computer Audit,” page 77
- 5.2,“System Installation Checklist,” page 79
- 5.3,“Supercon Shim Values,” page 81
- 5.4,“Liquids Probes Test Results,” page 83
- 5.5,“Console and Magnet Test Results,” page 87

Notes:

5.1 Computer Audit

Please provide the following information about your site (please print).

Company/University _____

Address _____

_____

Principal User _____

Phone _____ Spectrometer type _____

Fax _____ Console S/N _____

Sales Order No. _____ Delivery (month/day) _____

Please provide the following information for each computer. Additional forms are on the back of this page. Include computers directly attached to the spectrometer, computers (networked or non-networked, on-site or off-site) used to process NMR data using Varian's VNMR software, and computers (on-site and off-site) used to process data collected on this spectrometer with software from other vendors.

Information on computer ____ of ____ (e.g., 1 of 3)

Manufacturer _____ Model no. _____

Computer S/N _____ Purchased from _____

Memory (Mbytes) _____ Screen size (in.) _____

Peripherals: Internal hard disk (Mbytes) _____

External hard disk (Mbytes) _____ Serial no. _____

Tape drive size _____ Serial no. _____

CD-ROM drive model _____ Serial no. _____

Printer model _____ Serial no. _____

Plotter model _____ Serial no. _____

Terminal model _____ Serial no. _____

Other peripheral _____ Serial no. _____

Computer function: NMR host _____

Workstation running VNMR _____ on-site or off-site

Workstation running other NMR software _____ on-site or off-site

Workstation running VNMR and other NMR software _____ on-site or off-site

VNMR version _____ **Operating system** _____

The above computer audit was performed during installation of the system.

_____

Varian Representative Date

I certify that the information on this form is accurate and that all computers to be used to run VNMR software (including variants VnmrS, VnmrX, VnmrI, VnmrSGI, and VnmrV), or to run other software to process data obtained on this spectrometer, have been included in the audit (including those previously registered as part of purchases of other Varian NMR spectrometers).

_____

Customer Representative Date

Use these forms for additional computers. If more forms are needed, copy this page. Attach all copies to the Computer Audit.

Information on computer ____ of ____ (e.g., 2 of 3)

Manufacturer _____ Model no. _____

Computer S/N _____ Purchased from _____

Memory (Mbytes) _____ Screen size (in.) _____

Peripherals: Internal hard disk (Mbytes) _____

External hard disk (Mbytes) _____ Serial no. _____

Tape drive size _____ Serial no. _____

CD-ROM drive model _____ Serial no. _____

Printer model _____ Serial no. _____

Plotter model _____ Serial no. _____

Terminal model _____ Serial no. _____

Other peripheral _____ Serial no. _____

Computer function: NMR host _____

Workstation running VNMR _____ on-site or off-site

Workstation running other NMR software _____ on-site or off-site

Workstation running VNMR and other NMR software _____ on-site or off-site

VNMR version _____ **Operating system** _____

Information on computer ____ of ____ (e.g., 3 of 3)

Manufacturer _____ Model no. _____

Computer S/N _____ Purchased from _____

Memory (Mbytes) _____ Screen size (in.) _____

Peripherals: Internal hard disk (Mbytes) _____

External hard disk (Mbytes) _____ Serial no. _____

Tape drive size _____ Serial no. _____

CD-ROM drive model _____ Serial no. _____

Printer model _____ Serial no. _____

Plotter model _____ Serial no. _____

Terminal model _____ Serial no. _____

Other peripheral _____ Serial no. _____

Computer function: NMR host _____

Workstation running VNMR _____ on-site or off-site

Workstation running other NMR software _____ on-site or off-site

Workstation running VNMR and other NMR software _____ on-site or off-site

VNMR version _____ **Operating system** _____

5.2 System Installation Checklist

Company/University _____

Address _____

_____

Principal User _____

Phone _____ Spectrometer type _____

Fax _____ Console S/N _____

Sales Order No. _____ Magnet S/N _____

Shipment Damage:

_____

_____

Preinstallation Preparation:

Line voltage measured (Vac): console _____ accessory _____

Line pressure: air _____ N_2 _____

Air conditioning: _____

Cryogens (liters): LHe _____ LN _____

Testing and Customer Familiarization:

1. Acceptance tests and computer audit

   ___ Acceptance tests procedures finished

   ___ Test results form completed and signed

   ___ Computer audit completed and signed

2. System documentation review

   ___ *Software Object Code License Agreement* (acceptance of product constitutes acceptance of object code license regardless of whether agreement is signed or not)

   ___ Varian and OEM manuals

   ___ Explanation of warranty and where to telephone for information

3. Magnet demonstration

   ___ Posting requirements for magnetic field warning signs

   ___ Warning signs posted

   ___ Cryogenics handling and safety

   ___ Magnet refilling

   ___ Flowmeters

   ___ Homogeneity disturbances

4. Console and probe demonstration

   CAUTION! To avoid possible preamplifier damage, make sure the probe is connected and tuned to resonance.

   ___ Loading programs, operating the streaming tape unit

   ___ Experiment setup / using *GLIDE*

   ___ Using *GLIDE* for basic operation to obtain typical ^{1}H and ^{13}C spectra

   ___ Demonstration of APT and DEPT spectra

   ___ Demonstration of optional homonuclear and heteronuclear decoupling

Notes:

5.3 Supercon Shim Values

Fill in the following information for the supercon shims.

Magnet Frequency and Serial Number:

Magnet Frequency _____

Serial Number _____

Measurement in:

Helipot _____

Amps _____

Measurement	1. Date:	2. Date:	3. Date:
Z0			
Z1			
Z2			
Z3			
Z4			
X			
Y			
ZX			
ZY			
XY			
X2–Y2			
Drift			
Spacers			
Main Field Current			
Customer Signature:			
Varian Representative Signature:			

Notes

5.4 Liquids Probes Test Results

Please provide the following information about the probes and system. List any special hardware or conditions below under *Notes*. Then, fill in the results on the following sheets for every test performed on each probe.

Probe Size, Model, and Serial Number:

Probe Size and Model		Serial Number
Probe 1		
Probe 2		
Probe 3		
Probe 4		
Probe 5		

System Frequency:

____ 200 MHz ____

____ 300 MHz ____

____ 400 MHz ____

Notes:

Resolution and Lineshape (50%/0.55%/0.11%, Hz)

	^{1}H Spin ($CHCl_3$)	^{13}C Spin (ASTM)	Notes (Z0, lock gain, lock power values for D2O and acetone shims)
Probe 1			
Probe 2			
Probe 3			
Probe 4			
Probe 5			

Spinning Sidebands

	^{1}H	^{13}C	Notes
Probe 1			
Probe 2			
Probe 3			
Probe 4			
Probe 5			

90 Pulse Width (s)

	^{1}H	^{19}F	^{31}P	^{13}C	^{15}N	^{29}Si	Notes
Probe 1							
Probe 2							
Probe 3							
Probe 4							
Probe 5							

Sensitivity (S/N)

	^{1}H	^{19}F	^{31}P	^{13}C	^{15}N	^{29}Si	Notes (Z0, lock gain, lock power values for CDCl3, C6D6, and DMSO shims)
Probe 1							
Probe 2							
Probe 3							
Probe 4							
Probe 5							

γH_2 (Hz)

	γH_2 Specification	Notes
Probe 1		
Probe 2		
Probe 3		
Probe 4		
Probe 5		

VT Range (C)

	Temperature Range	Notes
Probe 1		
Probe 2		
Probe 3		
Probe 4		
Probe 5		

Other

	Specification	Notes
Probe 1		
Probe 2		
Probe 3		
Probe 4		
Probe 5		

Varian Representative Date

Customer Representative Date

Notes

5.5 Console and Magnet Test Results

GLIDE Operation Demonstration

_____

_____

APT and DEPT Demonstration

_____

_____

Homonuclear Decoupling (Optional)

_____

_____

Variable Temperature Operation (Optional)

_____

_____

Magnet Drift Test

_____

_____

_____
Varian Representative Date

_____
Customer Representative Date

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